UTILITY PATENT APPLICATION TRANSMITTAL

(Only for new nonprovisional applications under 37 CFR 1.53(b))

Attorney Docket No.	35.C10530 C1/D2
First Named Inventor or Application Identifier	
KIYOFUMI SAKAGUCHI, ET AL.	
Express Mail Label No.	

APPLICATION ELEMENTS
See MPEP chapter 600 concerning utility patent application contents.

ADDRESS TO: Assistant Commissioner for Patents
Box Patent Application
Washington, DC 20231

1. ☐ Fee Transmittal Form
(Submit an original, and a duplicate for fee processing)

2. ☒ Specification   *Total Pages* 60

3. ☐ Drawing(s) *(35 USC 113)*   *Total Sheets* 5

4. ☐ Oath or Declaration   *Total Pages* 2

   a. ☐ Newly executed (original or copy)

   b. ☐ Unexecuted for information purposes

   c. ☒ Copy from a prior application (37 CFR 1.63(d))
(for continuation/divisional with Box 17 completed)
[Note Box 5 below]

     i. ☐ DELETION OF INVENTOR(S)
Signed Statement attached deleting
inventor(s) named in the prior application,
see 37 CFR 1.63(d)(2) and 1.33(b).

5. ☐ Incorporation By Reference *(useable if Box 4c is checked)*
The entire disclosure of the prior application, from which a copy of
the oath or declaration is supplied under Box 4c, is considered as
being part of the disclosure of the accompanying application and is
hereby incorporated by reference therein.

6. ☐ Microfiche Computer Program *(Appendix)*

7. Nucleotide and/or Amino Acid Sequence Submission
(if applicable, all necessary)

   a. ☐ Computer Readable Copy

   b. ☐ Paper Copy (identical to computer copy)

   c. ☐ Statement verifying identity of above copies

ACCOMPANYING APPLICATION PARTS

8. ☐ Assignment Papers (cover sheet & document(s))

9. ☐ 37 CFR 3.73(b) Statement
(when there is an assignee)   ☐ Power of Attorney

10. ☐ English Translation Document *(if applicable)*

11. ☐ Information Disclosure Statement (IDS)/PTO-1449   ☐ Copies of IDS Citations

12. ☒ Preliminary Amendment

13. ☒ Return Receipt Postcard (MPEP 503)
(Should be specifically itemized)

14. ☐ Small Entity Statement(s)   ☐ Statement filed in prior application Status still proper and desired

15. ☐ Certified Copy of Priority Document(s)
(if foreign priority is claimed)

16. ☐ Other: _____

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17. If a CONTINUING APPLICATION, *check appropriate box and supply the requisite information:*

☐ Continuation   ☒ Divisional   ☐ Continuation-in-part (CIP)   of prior application No. __08/863,717__

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18. CORRESPONDENCE ADDRESS

☒ Customer Number or Bar Code Label   05514
(Insert Customer No. or Attach bar code label here)   or ☐ Correspondence address below

NAME	
Address	
City		State		Zip Code	
Country		Telephone		Fax	

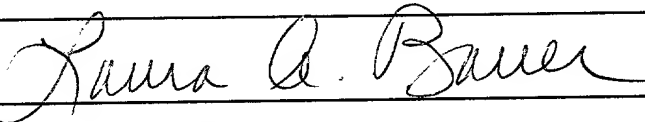| CLAIMS	(1) FOR	(2) NUMBER FILED	(3) NUMBER EXTRA	(4) RATE	(5) CALCULATIONS
	TOTAL CLAIMS (37 CFR 1.16(c))	75-20 =	55	X $ 22.00 =	$1,210.00
	INDEPENDENT CLAIMS (37 cfr 1.16(b))	3-3 =	0	X $ 82.00 =	$0
	MULTIPLE DEPENDENT CLAIMS (if applicable) (37 CFR 1.16(d))			$270.00 =	$0
				BASIC FEE (37 CFR 1.16(a))	$790.00
			Total of above Calculations =		$2,000.00
	Reduction by 50% for filing by small entity (Note 37 CFR 1.9, 1.27, 1.28).				
				TOTAL =	$2,000.00

19. Small entity status

   a. ☐ A Small entity statement is enclosed

   b. ☐ A small entity statement was filed in the prior nonprovisional application and such status is still proper and desired.

   c. ☐ Is no longer claimed.

20. ☒ A check in the amount of $ 2,000.00 _____ to cover the filing fee is enclosed.

21. ☐ A check in the amount of $ _____ to cover the recordal fee is enclosed.

22. The Commissioner is hereby authorized to credit overpayments or charge the following fees to Deposit Account No. 06-1205:

   a. ☒ Fees required under 37 CFR 1.16.

   b. ☒ Fees required under 37 CFR 1.17.

   c. ☐ Fees required under 37 CFR 1.18.

SIGNATURE OF APPLICANT, ATTORNEY, OR AGENT REQUIRED	
NAME	LAURA A. BAUER, Reg. No. 29,767
SIGNATURE	*Laura A. Bauer*
DATE	September 28, 1998

F501\A617003\ma

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)
                               :   Examiner: NYA
KIYOFUMI SAKAGUCHI ET AL.   )
                               :
Divisional of   )   Group Art Unit: NYA
Application No.: 08/863,717   :
)
Filed: Herewith   :
)
For: PROCESS FOR PRODUCTION   :
      OF SEMICONDUCTOR   )
      SUBSTRATE   :   September 28, 1998

PRELIMINARY AMENDMENT

Assistant Commissioner for Patents
Washington, D.C.   20231

Sir:

Preliminary to the examination on the merits,
please amend the above-identified application as follows:

IN THE CLAIMS:

Please cancel claims 1-21 without prejudice or
disclaimer.

Please add the following new claims 22-96:

--22.   A process for producing a semiconductor
substrate comprising the steps of:

preparing a first substrate having a nonporous monocrystalline semiconductor layer on a monocrystalline semiconductor substrate with the interposition of a porous layer, said first substrate having two opposing faces;

bonding a second substrate, said second substrate having two opposing faces, to said first substrate to yield a multilayer structure where said nonporous monocrystalline semiconductor layer is arranged inside; and

separating the multilayer structure at said porous layer such that said nonporous monocrystalline semiconductor layer remains on said second substrate.

23. The process according to claim 25, wherein said step of separating said multilayer structure is conducted by applying a tensile force to said multilayer structure in a direction perpendicular to the faces of said first and second substrates.

24. The process according to claim 25, wherein said step of separating said multilayer structure is conducted by applying a compression force to said multilayer structure in a direction perpendicular to the faces of said first and second substrates.

25. The process according to claim 25, wherein said step of separating said multilayer structure is conducted by applying a shearing force to said multilayer

structure in a direction parallel to the faces of said first and second substrates.

26. The process according to claim 25, wherein said step of separating said multilayer structure is conducted by applying a wave energy to said multilayer structure to break said porous layer.

27. The process according to claim 29, wherein said wave energy is ultrasonic.

28. The process according to claim 25, wherein said step of separating said multilayer structure is conducted by inserting a sharp blade into said porous layer of said multilayer structure.

29. The process according to claim 25, wherein said step of separating said multilayer structure is conducted by impregnating a liquid into said porous layer of said multilayer structure, followed by heating or cooling said multilayer structure such that the liquid expands.

30. The process according to claim 25, wherein said step of separating said multilayer structure is conducted by beginning to etch selectively said porous layer at the edge of said multilayer structure.

31.   The process according to claim 25, wherein said step of separating said multilayer structure is conducted such that a portion of said porous layer remains together with said nonporous monocrystalline semiconductor layer on said second substrate and further comprises removing the portion of said porous layer from said second substrate where the separation has been carried out.

32.   The process according to claim 34, wherein said step of removing the portion of said porous layer is conducted by etching the portion of said porous layer on said second substrate such that said nonporous monocrystalline semiconductor layer remains.

33.   The process according to claim 34, wherein said step of removing the portion of said porous layer is conducted by grinding the portion of said porous layer on said second substrate such that said nonporous monocrystalline semiconductor layer remains.

34.   The process according to claim 25, wherein said step of separating said multilayer structure is conducted such that a portion of said porous layer is not removed from said semiconductor substrate, followed by removing the portion of said porous layer remaining on said semiconductor substrate.

35. The process according to claim 37, wherein said process is further comprised of flattening a surface of said monocrystalline semiconductor substrate where the portion of said porous layer has been removed.

36. The process according to claim 25, wherein said first substrate is formed by making a portion of a silicon substrate porous to form a porous layer, and epitaxially growing a nonporous monocrystalline semiconductor layer on said porous layer.

37. The process according to claim 39, wherein said silicon substrate portion is made porous by anodization.

38. The process according to claim 40, wherein current density is altered in stages when said anodization is carried out.

39. The process according to claim 39, wherein said epitaxial growth is conducted by a method selected from a group consisting of a molecular beam epitaxy method, a plasma CVD method, a reduced pressure CVD method, a photo-assisted CVD method, a bias sputtering method, and a liquid-phase epitaxial growth method.

40.	The process according to claim 25, wherein said nonporous monocrystalline semiconductor layer is comprised of a monocrystalline silicon layer.

41.	The process according to claim 25, wherein said nonporous monocrystalline semiconductor layer is comprised of a monocrystalline compound semiconductor layer.

42.	The process according to claim 44, wherein said monocrystalline compound semiconductor layer is comprised of a GaAs layer or an AlGaAs layer.

43.	The process according to claim 25, wherein said porous layer is comprised of plural layers of different porosity.

44.	The process according to claim 25, wherein said first and second substrates are bonded through an insulating layer.

45.	The process according to claim 47, wherein said insulating layer is comprised of an oxidized film formed by oxidizing a surface of said nonporous monocrystalline semiconductor layer.

46.   The process according to claim 47, wherein said insulating layer is comprised of an insulating thin plate, and said first and second substrates are bonded with the interposition of said plate to form a three layer structure.

47.   The process according to claim 47, wherein said insulating layer is comprised of a SiO_2 layer formed on said second substrate.

48.   The process according to claim 25, wherein said second substrate is comprised of a silicon substrate.

49.   The process according to claim 25, wherein said second substrate is a light transmissive substrate.

50.   The process according to claim 52, wherein said second substrate is a quartz substrate or a glass substrate.

51.   A process for producing a semiconductor substrate comprising the steps of:

preparing a first substrate having two nonporous monocrystalline semiconductor layers located on the respective sides of a monocrystalline semiconductor with the interposition of respective porous layers, said first substrate having two opposing faces;

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bonding two second substrates to the
respective faces of said first substrate to yield a
multilayer structure, each of said second substrates having
two opposing faces; and

separating the multilayer structure at each of
said two porous layers such that said two nonporous
monocrystalline semiconductor layers exist on each of said
respective two second substrates.

52. The process according to claim 54, wherein
said step of separating said multilayer structure is
conducted by applying a tensile force to said multilayer
structure in a direction perpendicular to the faces of said
first substrate and said two second substrates.

53. The process according to claim 54, wherein
said step of separating said multilayer structure is
conducted by impregnating a liquid into each of said two
porous layers of said multilayer structure, followed by
heating or cooling said multilayer structure to expand the
liquid.

54. The process according to claim 54, wherein
said step of separating said multilayer structure is
conducted such that a portion of said porous layer remains
together with said nonporous monocrystalline semiconductor

layer on each of said two second substrates and further comprises removing the portion of said porous layer from each of said two second substrates where the separation has been carried out.

55. The process according to claim 57, wherein said step of removing the portion of said porous layer is conducted by etching the portion of said porous layer on said two second substrates such that said nonporous monocrystalline semiconductor layer remains.

56. The process according to claim 57, wherein said step of removing the portion of said porous layer is conducted by grinding the portion of said porous layer on said two second substrates such that said nonporous monocrystalline semiconductor layer remains.

57. The process according to claim 54, wherein said step of separating said multilayer structure is conducted such that a portion of said porous layer is not removed from each side of said monocrystalline semiconductor substrate, followed by removing the portion of said porous layers remaining on both sides of said monocrystalline semiconductor substrate.

58. The process according to claim 60, wherein said process is further comprised of flattening respective surfaces of both sides of said monocrystalline semiconductor substrate where the portion of said porous layer has been removed.

59. The process according to claim 54, wherein said first substrate is formed by making both sides of a silicon substrate partially porous to form two porous layers, and subsequently epitaxially growing two nonporous monocrystalline semiconductor layers on each said two porous layers.

60. The process according to claim 62, wherein both sides of the silicon substrate are made porous by anodization.

61. The process according to claim 62, wherein said epitaxial growth is conducted by a method selected from a group consisting of a molecular beam epitaxy method, a plasma CVD method, a reduced pressure CVD method, a photo-assisted CVD method, a bias sputtering method, and a liquid-phase growth method.

62. The process according to claim 54, wherein said two nonporous monocrystalline semiconductor layers are comprised of two monocrystalline silicon layers.

63. The process according to claim 54, wherein said two second substrates are bonded to said first substrate through two insulating layers.

64. The process according to claim 66, wherein each of said two insulating layers in comprised of an oxidized film formed by oxidizing a surface of each of said two nonporous monocrystalline semiconductor layers.

65. The process according to claim 66, wherein each of said two insulating layers is comprised of an insulating thin plate, and said two second substrates are bonded to said first substrate with the interposition of said two insulating thin plates, to form a five layer structure.

66. The process according to claim 66, wherein each of said two insulating layers is comprised of a SiO_2 layer formed on each of said two second substrates.

67. The process according to claim 54, wherein said second substrate is comprised of a silicon substrate.

68. A process for producing a semiconductor
substrate comprising the steps of:

preparing a first substrate having a
monocrystalline semiconductor layer on a monocrystalline
semiconductor substrate with the interposition of a layer
having a lower mechanical strength than that of said
monocrystalline semiconductor substrate, said first substrate
having two opposing faces;

bonding a second substrate to said first
substrate to yield a multilayer structure where said
monocrystalline semiconductor layer is arranged inside, said
second substrate having two opposing faces; and

separating the multi-layer structure at said
layer having the lower mechanical strength such that said
monocrystalline semiconductor layer exists on said second
substrate.

69. The process according to claim 71, wherein
said step of separating said multilayer structure is
conducted by applying a tensile force to said multilayer
structure in a direction perpendicular to the faces of said
first and second substrates.

70. The process according to claim 71, wherein
said step of separating said multi-layer structure is
conducted by applying a compression force to said multi-layer

structure in a direction perpendicular to the faces of said first and second substrates.

71. The process according to claim 71, wherein said step of separating said multilayer structure is conducted by applying a shearing force to said multilayer structure in a direction parallel to the faces of said first and second substrates.

72. The process according to claim 71, wherein said step of separating said multilayer structure is conducted by applying a wave energy to said multilayer structure to break said layer having the lower mechanical strength.

73. The process according to claim 75, wherein said wave energy is ultrasonic.

74. The process according to claim 71, wherein said step of separating said multilayer structure is conducted by inserting a sharp blade into said layer having the lower mechanical strength of said multilayer structure.

75. The process according to claim 71, wherein said step of separating said multilayer structure is conducted by impregnating a liquid into said layer having the

lower mechanical strength of said multilayer structure,
followed by heating or cooling said multi-layer structure to
expand the liquid.

76. The process according to claim 71, wherein
said step of separating said multilayer structure is
conducted by beginning to etch selectively said layer having
the lower mechanical strength at the edge of said multilayer
structure.

77. The process according to claim 71, wherein
said step of separating said multilayer structure is
conducted such that a portion of said layer having the lower
mechanical strength remains together with said
monocrystalline semiconductor layer on said second substrate
and further comprises removing the portion of said layer
having the lower mechanical strength from said second
substrate where the separation has been carried out.

78. The process according to claim 80, wherein
said step of removing the portion of said layer having the
lower mechanical strength is conducted by etching the portion
of said layer having the lower mechanical strength above said
second substrate such that said monocrystalline semiconductor
layer remains.

79. The process according to claim 80, wherein said step of removing the portion of said layer having the lower mechanical strength is conducted by grinding the portion of said layer having the lower mechanical strength above said second substrate such that said monocrystalline semiconductor layer remains.

80. The process according to claim 71, wherein said step of separating said multilayer structure is conducted such that a portion of said layer having the lower mechanical strength is not removed from said semiconductor substrate, followed by removing the portion of said layer having the lower mechanical strength remaining on said semiconductor substrate.

81. The process according to claim 83 wherein said process further comprises flattening a surface of said monocrystalline semiconductor substrate where the portion of said layer having the lower mechanical strength has been removed.

82. The process according to claim 71, wherein said first substrate is formed by making a portion of a silicon substrate porous to form a layer having the lower mechanical strength, and epitaxially growing a

monocrystalline semiconductor layer on said layer having the lower mechanical strength.

83. The process according to claim 85, wherein the silicon substrate portion is made porous by anodization.

84. The process according to claim 86, wherein current density is altered in stages when said anodization is carried out.

85. The process according to claim 85, wherein said epitaxial growth is conducted by a method selected from a group consisting of a molecular beam epitaxy method, a plasma CVD method, a reduced pressure CVD method, a photo-assisted CVD method, a bias sputtering method, and a liquid-phase growth method.

86. The process according to claim 71, wherein said monocrystalline semiconductor layer is comprised of a monocrystalline silicon layer.

87. The process according to claim 71, wherein said monocrystalline semiconductor layer is comprised of a monocrystalline compound semiconductor layer.

88. The process according to claim 90, wherein said monocrystalline compound semiconductor layer is comprised of a GaAs layer or an AlGaAs layer.

89. The process according to claim 71, wherein said layer having the lower mechanical strength is comprised of plural layers of different porosity.

90. The process according to claim 71, wherein said first and second substrates are bonded through an insulating layer.

91. The process according to claim 93, wherein said insulating layer is comprised of an oxidized film formed by oxidizing a surface of said monocrystalline semiconductor layer.

92. The process according to claim 93, wherein said insulating layer is comprised of an insulating thin plate, and said first and second substrates are bonded with the interposition of said plate to form a three layer structure.

93. The process according to claim 93, wherein said insulating layer is comprised of a SiO_2 layer formed on said second substrate.

94. The process according to claim 71, wherein said second substrate is comprised of a silicon substrate.

95. The process according to claim 71, wherein said second substrate is a light transmissive substrate.

96. The process according to claim 98, wherein said second substrate is a quartz substrate or a glass substrate.--

REMARKS

As indicated above, this application is a divisional of Application No. 08/863,717. Claims 22-96 are now presented for examination in this application. Claims 1-21 filed with the parent application have been cancelled without prejudice or disclaimer of subject matter. Claims 22, 51 and 68 are now the only independent claims.

Applicants note that claims 22-96 which are now presented for examination are the same as the claims 25-99 Applicant filed in the parent case Application No. 08/863,717 in a Preliminary Amendment dated July 28, 1997.

Since a substitute specification had been filed in the parent case, a copy of the application as originally filed, as well as the substitute specification is included with this filing.

Applicants' undersigned attorney may be reached in our New York office by telephone at (212) 218-2100. All correspondence should continue to be directed to our below listed address.

Respectfully submitted,

Laura A. Bauer

Attorney for Applicants

Registration No. _39,767_

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TITLE

PROCESS FOR PRODUCTION OF SEMICONDUCTOR SUBSTRATE

BACKGROUND OF THE INVENTION

Field of the Invention

The present invention relates to a process for
producing a semiconductor substrate. More
specifically, the present invention relates to a
process for producing a monocrystalline semiconductor
on a dielectric-isolated or insulative material, or a
monocrystalline compound semiconductor on a
semiconductor substrate. Further the present invention
relates to a process for producing an electronic device
or an integrated circuit formed on a single crystalline
semiconductor layer.

Related Background Art

The technique of formation of monocrystalline Si
(silicon) semiconductor on an insulative material is
well known as silicon-on-insulator (SOI) technique. A
device prepared by the SOI technique has various
advantages which are not achievable by a bulk Si
substrate in usual Si integrated circuits, as noted

1.   Ease of dielectric isolation, and possibility of high degree of integration'

2.   High resistance against radioactive ray;

3.   Low floating capacity, and the possibility of high speed operation;

4.   The welling process is unnecessary;

5.   Preventability of latch-up; and

6.   Possibility of producing a complete depletion type field-effect transistor to name a few.

The process of forming the SOI structure has been actively studied for several decades. The results of the studies are summarized, for example, in the paper: Special Issue; "Single-crystal silicon on non-single-crystal insulators"; edited by G.W. Cullen, Journal of Crystal Growth; Vol.63, No.3, pp.429-590 (1983).

SOS (silicon on sapphire) is known and is produced by heteroepitaxial growth of silicon on monocrystalline sapphire by CVD (chemical vapor deposition). The SOS technique, which is successful as one of SOI techniques, is limited in its application, because of many crystal defects caused by mismatch of the lattice at the interface between the Si layer and the underlying sapphire, contamination of the Si layer with aluminum from the sapphire substrate, expense of the substrate, and the difficulty of large-area substrate formation.

Recently, studies are being made to produce the SOI structure without using a sapphire substrate. The

studies are classified roughly into the two processes
below:

1. A first process which includes surface oxidation of
a monocrystalline Si substrate, local exposure of the
Si substrate by opening a window, and epitaxial growth
of Si laterally from the exposed portion as the seed to
form an Si layer on SiO_2. (Si layer deposition on
SiO_2).

2. A second process including SiO_2 formation beneath a
monocrystalline SiO_2 substrate, utilizing the SiO_2
substrate as the active layer. (No Si layer
deposition)

The device formed on a compound semiconductor exhibits
performances, such as high speed, and luminescence,
which are not achievable by Si. Such types of devices
are formed by epitaxial growth on a compound
semiconductor substrate such as GaAs. The compound
semiconductor substrate, however, has disadvantages of
high cost, low mechanical strength, and difficulty in
the formation of a large-area wafer. Accordingly,
heteroepitaxial growth of a compound semiconductor on
an Si wafer is being studied to attain low cost, high
mechanical strength, and ease of production of a
large-area wafer.

The above-known process 1 (Si layer deposition on SiO_2)
includes methods of direct lateral epitaxial growth of
monocrystalline Si layer by CVD; deposition of
amorphous Si and subsequent heat treatment to cause
solid-phase lateral epitaxial growth; melting
recrystallization to grow monocrystalline layer on an
SiO_2 by irradiation of amorphous or polycrystalline Si
layer with a focused energy beam such as an electron
beam and laser beam; and a zone melting

recrystallization in which a bar-shaped heater is moved
to scan with a belt-like melt zone. These methods
respectively have advantages and disadvantages,
involving problems in process controllability,
productivity, product uniformity, and product quality,
and are not industrialized yet. For example, the CVD
method requires sacrificial oxidation, giving low
crystallinity in the solid-phase growth. The beam
annealing method involves problems in processing time
of focused beam scanning and in controllability of beam
superposition and focusing. Of the above methods, the
zone melting recrystallization is the most advanced
method, and is employed in relatively large scale
integrated circuits. This method, however, still
causes crystal defects in subgrain boundaries, and is
not successful in the production of a minority carrier
device.

The above known process 2 in which the Si substrate is
not utilized as the seed for epitaxial growth includes
the four methods below:

1. An oxidation film is formed on a monocrystalline Si
substrate which has V-shaped grooves on the surface
formed by anisotropical etching; a polycrystalline Si
layer is deposited in a thickness approximate to that
of the Si substrate on the oxidation film; and the back
face of the Si substrate is ground to form a
monocrystalline Si region isolated dielectrically by
surrounding with the V-shaped grooves. This method
involves problems in controllability and productivity
in deposition of polycrystalline Si in a thickness of
as large as several hundred microns, and in removal of
the monocrystalline Si substrate by grinding at the
back face to leave an isolated active Si layer only.

2. An SiO$_2$ layer is formed by ion implantation into a
monocrystalline Si substrate (SIMOX: Separation by ion
implanted oxygen). This is the most highly advanced
method in view of the matching with the Si process.
This method, however, requires implantation of oxygen
ions in an amount of as much as 10^{18} ions/cm^2, which
takes a long time, resulting in low productivity and
high wafer cost. Further, the product has many
remaining crystal defects, and does not have
satisfactory properties for the industrial production
of a minority carrier device.

3. An SOI structure is formed by oxidation of porous
Si for dielectric isolation. In this method, an N-type
Si layer is formed in an island-like pattern on a
P-type monocrystalline Si substrate surface by proton
ion implantation (Imai, et al.: J. Crystal Growth, Vol.
63, p. 547 (1983)) or by epitaxial growth and
patterning, and subsequently only the P-type Si
substrate is made porous by anodic oxidation in an HF
solution to surround the island-patterned N-type Si,
and the N-type Si island is dielectrically isolated by
accelerated oxidation. In this method, the isolated Si
regions are fixed prior to the device process, which
may limit the freedom of device design
disadvantageously.

4. Different from the above conventional SOI
formation, a method has recently come to be noticed in
which a monocrystalline Si substrate is bonded to
another thermally oxidized monocrystalline Si substrate
by heat treatment or use of an adhesive to form an SOI
structure. This method requires uniform thinness of
the active layer for the device: namely, formation of a
film of a micron thick or thinner from a
monocrystalline substrate of several hundred microns

thick. This thin film may be formed by either of the
two methods below.

1.   Thin film formation by grinding; and

2.   Thin film formation by selective etching.

The grinding method does not readily give a
uniform thin film. In particular, formation of a film
of submicron thickness results in thickness variation
of tens of percent. This irregularity is a serious
problem. With a larger diameter of the wafer, the
uniformity of the thickness is much more difficult to
attain.

The etching method is regarded to be effective for
uniform thin film formation. This method, however,
involves the problems of insufficient selectivity of
about 10^2 at the highest, inferior surface properties
after etching, and low crystallinity of the SOI layer
because of the employed ion implantation, epitaxial or
heteroepitaxial growth on a high-concentration B-doped
Si layer. (C. Harendt, et al.: J. Elect. Mater., Vol.
20, p. 267 (1991); H. Baumgart, et al.: Extended
Abstract of ECS 1st International Symposium of Wafer
Bonding, pp. 733 (1991); and C.E. Hunt: Extended
Abstract of ECS 1st International Symposium of Wafer
Bonding, pp. 696(1991))

The semiconductor substrate which is prepared by
lamination requires two wafers essentially, and a major
part of one of the wafers is discarded by grinding or
etching, thereby wasting the resource. Therefore, the
SOI prepared by lamination involves many problems in
controllability, uniformity, production cost, and so
forth in conventional processes.

A thin Si layer deposited on a light-transmissive substrate typified by a glass plate becomes amorphous or polycrystalline owing to disorder of crystallinity of the substrate, not giving high performance of the device. Simple deposition of Si does not give desired quality of single crystal layer owing to the amorphous crystal structure of the substrate.

The light-transmissive substrate is essential for construction of a light-receiving element such as a contact sensor, and projection type of liquid crystal image-displaying apparatus. Additionally, a driving element of high performance is necessary for higher density, higher resolution, and higher precision of the sensor and of the image elements of the display. Consequently, the element provided on a light transmissive substrate is also required to have a monocrystalline layer of high crystallinity.

Amorphous Si or polycrystalline Si will not give a driving element having the required sufficient performance because of the many defects in the crystal structure.

As mentioned above, a compound semiconductor device requires essentially a compound semiconductor substrate. The compound semiconductor substrate, however, is expensive, and is not readily formed in a larger size.

Epitaxial growth of a compound semiconductor such as GaAs on an Si substrate gives a grown film of poor crystallinity owing to the difference in the lattice constants and the thermal expansion coefficients, thereby the resulting grown film being unsuitable for use for a device.

Epitaxial growth of a compound semiconductor on porous
Si is intended for mitigation of mismatch of the
lattices. However, the substrate does not have
sufficient stability and reliability owing to the low
thermal stability and long-term deterioration of the
porous Si.

In view of the above-mentioned problems, Takao
Yonehara, one of the inventors of the present
invention, disclosed formerly a novel process for
preparing a semiconductor member in European Patent
Publication No. 0469630A2. This process comprises the
steps of forming a member having a nonporous
monocrystalline semiconductor region on a porous
monocrystalline semiconductor region; bonding the
surface of a member of which the surface is constituted
of an insulating substance onto the surface of the
nonporous monocrystalline semiconductor region; and
then removing the porous monocrystalline semiconductor
region by etching. This process is satisfactory for
solving the above-mentioned problems. Further
improvement of the disclosed process for higher
productivity and lower production cost will contribute
greatly to the industries concerned.

SUMMARY OF THE INVENTION

The present invention intends to improve further the
process disclosed in the above European Patent for
producing a semiconductor member.

The present invention further intends to provide a
process for producing economically a semiconductor
substrate having a monocrystalline layer or a compound
semiconductor monocrystalline layer having excellent
crystallinity, large-area and a uniform flat surface on
a surface of a monocrystalline substrate, in which the

substrate is removed to leave the active semiconductor
layer to obtain a monocrystalline layer or a compound
semiconductor monocrystalline layer formed on the
surface and having few defects.

The present invention still further intends to provide
a process for producing a semiconductor substrate on a
transparent substrate (light-transmissive substrate)
for obtaining a monocrystalline Si semiconductor layer
or a monocrystalline compound semiconductor layer
having crystallinity as high as that of a
monocrystalline wafer with high productivity, high
uniformity, high controllability, and low production
cost.

The present invention still further intends to provide
a process for producing a semiconductor substrate
useful in place of expensive SOS or SIMOX in the
production of a large scale integrated circuit of SOI
structure.

A first embodiment of the process for producing a
semiconductor substrate of the present invention
comprises the steps of: forming a nonporous
monocrystalline semiconductor layer on a porous layer
of the first substrate having the porous layer; bonding
the nonporous monocrystalline layer onto a second
substrate; separating the bonded substrates at the
porous layer; removing the porous layer on the second
substrate; and removing the porous layer constituting
the first substrate.

A second embodiment of the process for producing a
semiconductor substrate of the present invention
comprises the steps of: forming a nonporous
monocrystalline semiconductor layer on a porous layer
of a first substrate having the porous layer; bonding

the nonporous monocrystalline layer onto a second substrate with interposition of an insulative layer; separating the bonded substrates at the porous layer; removing the porous layer on the second substrate; and removing the porous layer constituting the first substrate.

In the present invention, the lamination-bonded substrates are separated at the porous layer, and the porous layer is removed from the second substrate having a nonporous monocrystalline semiconductor layer. Thereby, a semiconductor substrate is prepared which has nonporous monocrystalline semiconductor layer of high quality. Furthermore, the first substrate can be repeatedly used for producing the semiconductor substrate in the next production cycle by removing the remaining porous layer on the first substrate after the separation of the two substrates. Thereby, the semiconductor substrate can be produced with higher productivity and lower cost.

The present invention enables preparation of a monocrystalline layer of Si or the like, or a monocrystalline compound semiconductor layer having excellent crystallinity similar to monocrystalline wafers on a substrate including a light-transmissive substrate with advantages in productivity, uniformity, controllability, and production cost.

The present invention further enables production of a semiconductor substrate which can be a substitute for expensive SOS and SIMOX in the production of large scale integrated circuits of an SOI structure.

According to the present invention, the combined substrates are separated at the porous layer or layers into two or more substrates, and the one or more

separated substrates may be used as a semiconductor substrate after removal of the remaining porous layer, and the other substrate may be used repeatedly in the next production cycle of a semiconductor substrate.

Further, according to the present invention, two semiconductor substrates can be produced simultaneously by forming porous layers and nonporous monocrystalline layers on the both faces of a substrate, bonding thereto two other substrates, and separating the substrates at the porous layer.

BRIEF DESCRIPTION OF THE DRAWINGS

Figs. 1A to 1E are schematic drawings for explaining an example of the process of the present invention.

Figs. 2A to 2E are schematic drawings for explaining another example of the process of the present invention.

Figs. 3A to 3E are schematic drawings for explaining a still another example of the process of the present invention.

Figs. 4A to 4E are schematic drawings for explaining a further example of the process of the present invention.

Figs. 5A to 5E are schematic drawings for explaining a still further example of the process of the present invention.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The process for producing a semiconductor substrate of
the present invention is described by employing a
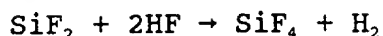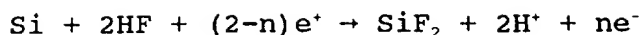silicon substrate as an example.

The mechanical strength of porous silicon is much lower
than that of bulk silicon depending on the porosity
thereof. For instance, porous silicon having a
porosity of 50% is considered to have half the
mechanical strength of bulk silicon. Therefore, on
application of a tensile force, a compressive force, or
a shearing force to a laminated wafer, the porous layer
will be broken first. The larger the porosity of the
porous layer, the less force is needed for the
breakdown of the layer.

A silicon substrate can be made porous by anodization
in an HF solution. The resulting porous Si layer has a
density ranging from 1.1 to 0.6 g/cm^3 depending on the
HF solution concentration of from 50 to 20% in
comparison with the density of 2.33 g/cm^3 of
monocrystalline Si. The porous layer is formed only on
a P-type Si substrate, but is not formed on an N-type
Si layer for the reasons described later. The porous
Si layer has pores of about 600 Å in average diameter
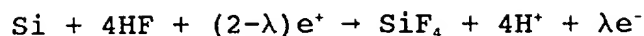according to transmissive electron microscopy.

The porous Si was found by Uhlir, et al. in the year
1956 during the study of electropolishing of
semiconductors (A. Uhlir: Bell Syst. Tech. J., vol. 35,
p. 333 (1956)).

Unagami, et al. found that positive holes are required
for anodization of Si in an HF solution, and the
reactions proceed as shown in their report on
dissolution of Si in anodization (T. Unagami, et al.:
J. Electrochem. Soc., vol. 127, p. 476 (1980)) as
below:

$$Si + 2HF + (2-n)e^+ \rightarrow SiF_2 + 2H^+ + ne^-$$
$$SiF_2 + 2HF \rightarrow SiF_4 + H_2$$
$$SiF_4 + 2HF \rightarrow H_2SiF_6$$

or

$$Si + 4HF + (2-\lambda)e^+ \rightarrow SiF_4 + 4H^+ + \lambda e^-$$
$$SiF_4 + 2HF \rightarrow H_2SiF_6$$
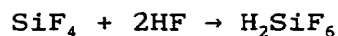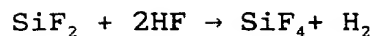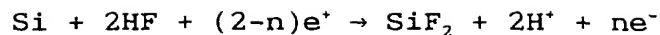
where e_+ and e_- represent respectively a positive hole and an electron; n and λ represent respectively the number of positive holes required for dissolving one Si atom. Unagami reported that porous Si is formed under the condition of n > 2, or λ > 4.

According to the above consideration, P-type Si which has positive holes can be made porous, whereas N-type Si cannot be made porous. This selectivity for porosity was evidenced by Nagano, et al., and Imai (Nagano, Nakajima, Yasuno, Oonaka, and Kajihara: Denshi Tsushin Gakkai Gijutsu Kenkyu Hokoku (Technical Research Report of Electronic Communication Society) vol. 79, SSD79-9549 (1979); and K. Imai: Solid-State Electronics, vol. 24, p. 159 (1981)).

On the other hand, a report is found that high concentration N-type Si can be made porous (R.P. Holmstrom and J.Y. Chi: Appl. Phys. Lett., vol. 42, p. 386 (1983)). Therefore, selection of the substrate is important for producing porous Si regardless of P-type or N-type.

The porous Si layer has pores of about 600 Å in average diameter by observation by transmission electron microscopy, and the density is less than half that of monocrystalline Si. Nevertheless, the single crystallinity is maintained, and thereon a monocrystalline Si can be made to grow epitaxially in a layer. However, in the epitaxial growth at a temperature of 1000°C or higher, the internal pores

will come to be rearranged, which impairs the
accelerated etching characteristics. Therefore, low
temperature growth processes are preferred for
epitaxial growth of the Si layer, such as molecular
beam epitaxial growth, plasma CVD, reduced pressure
CVD, photo-assisted CVD, bias sputtering, and liquid-
phase epitaxial growth.

The porous layer has a large volume of voids therein,
having a half or lower density of the material, and
having a surface area remarkably large for the volume.
Accordingly, the chemical etching is greatly
accelerated in comparison with that of the normal
monocrystalline layer.

Embodiment 1

A first monocrystalline Si substrate 11 is made porous
at the surface to form a porous layer 12 as shown in
Fig. 1A. Then, nonporous monocrystalline Si layer 13
is formed on the porous Si layer 12 as shown in Fig.
1B.

Another Si supporting substrate 14 is brought into
contact with the nonporous monocrystalline Si layer 13
with interposition of an insulative layer 15 at room
temperature as shown in Fig. 1C, and then the contacted
matter was subjected to anode coupling, compression,
heat treatment, or combination thereof to bond tightly
the Si supporting substrate 14 and the monocrystalline
layer 13 with interposition of the insulative layer 15.
The insulative layer 15 may be formed preliminarily on
either one of the monocrystalline Si layer 13 or the Si
supporting substrate 14, or the three sheets may be
bonded with an insulative thin film interposed.

Subsequently, the substrates are separated at the porous Si layer 12 as shown in Fig. 1D. On the Si supporting substrate 14, the layers have the structure of porous Si 12 / monocrystalline Si layer 13 / insulative layer 15 / Si supporting substrate 14.

The porous Si 12 is removed selectively by non-electrolytic wet chemical etching by use of at least one of a usual Si etching solution, hydrofluoric acid or a mixture of hydrofluoric acid with alcohol and/or hydrogen peroxide as the porous Si-selective etching solution, and buffered hydrofluoric acid or a mixture of hydrofluoric acid with alcohol and/or hydrogen peroxide to leave the thin-layered monocrystalline Si layer 13 on the insulative substrate 15 and 14. As described above in detail, the porous Si can be etched selectively by a usual Si etching solution owing to the extremely large surface area of the porous surface area.

Otherwise, the porous Si 12 is selectively removed by grinding by utilizing the monocrystalline Si layer 13 as the grinding stopper.

Fig. 1E illustrates a semiconductor substrate of the present invention. The monocrystalline Si layer 13 is formed flat and uniformly in a thin layer on the insulative substrate 15 and 14 over the entire large area of the wafer. The resulting semiconductor substrate is useful for production of insulation-isolated electronic elements.

The first monocrystalline Si substrate 11 may be repeatedly used for the same use after removal of the remaining Si and surface flattening treatment if the surface has become roughened unacceptably in the next production cycle.

The method of separation of the two substrates at the porous Si layer in the present invention includes crushing of the porous layer by compression on both faces of the bonded substrates; pulling of the respective substrates in opposite directions; insertion of a jig or the like into the porous layer; application of force in opposite directions parallel to the bonded face of the substrates; application of supersonic vibration to the porous layer; and so forth.

The porosity of the porous Si layer suitable for the separation ranges generally from 10 to 80%, preferably from 20 to 60%.

Embodiment 2

A first monocrystalline Si substrate 21 is made porous at the surface to form a porous layer 22 as shown in Fig. 2A. Then a nonporous monocrystalline Si layer 23 is formed on the porous Si layer 22 as shown in Fig. 2B.

A light-transmissive supporting substrate 24 is brought into contact with the monocrystalline Si layer 23 with interposition of an insulative layer 25 at room temperature as shown in Fig. 2C, and then the contacted matter was subjected to anode coupling, compression, heat treatment, or combination of the treatment to bond tightly the light-transmissive supporting substrate 24 and the monocrystalline layer 23 with interposition of the insulative layer 25. The insulative layer 25 may be formed preliminarily on either one of the monocrystalline Si layer or the light-transmissive supporting substrate 24, or the three sheets may be bonded with interposition of an insulative thin film.

Subsequently, the substrates are separated at the porous Si layer 22 as shown in Fig. 2D. On the light-transmissive supporting substrate, the layers have the structure of porous Si 22 / monocrystalline Si layer 23 / insulative layer 25 / light-transmissive supporting substrate 24.

The porous Si 22 is removed selectively by non-electrolytic wet chemical etching by use of at least one of a usual Si etching solution, hydrofluoric acid or a mixture of hydrofluoric acid with alcohol and/or hydrogen peroxide as the porous Si-selective etching solution, and buffered hydrofluoric acid or a mixture of hydrofluoric acid with alcohol and/or hydrogen peroxide to leave a thin-layered monocrystalline Si layer 23 on the insulative substrate 25 and 24. As described above in detail, the porous Si can be etched selectively by a usual Si etching solution because of the extremely large surface area of the porous surface area.

Otherwise, the porous Si 23 is selectively removed by grinding by utilizing the monocrystalline Si layer 22 as the grinding stopper.

Fig. 2E illustrates a semiconductor substrate of the present invention. The monocrystalline Si layer 23 is formed flat and uniformly in a thin layer on the insulative substrate 25 and 24 over the entire large area of the wafer. The obtained semiconductor substrate is useful for production of insulation-isolated electronic elements.

The presence of the interposed insulative layer 25 is not essential.

The first monocrystalline Si substrate 21 may be repeatedly used for the same use after removal of the remaining Si and surface flattening treatment if the surface has become roughened unacceptably in the next production cycle.

Embodiment 3

A first monocrystalline Si substrate 31 is made porous at the surface to form a porous layer 32 as shown in Fig. 3A. Then a nonporous monocrystalline compound semiconductor layer 33 is formed on the porous Si layer 32 as shown in Fig. 3B.

Another Si supporting substrate 34 is brought into close contact with the monocrystalline compound semiconductor layer 33 with interposition of an insulative layer 35 at room temperature as shown in Fig. 3C, and then the contacted matter was subjected to anode coupling, compression, or heat treatment, or combination of the treatments to bond tightly the Si supporting substrate 34 and the monocrystalline layer 33 with interposition of the insulative layer 35. The insulative layer 35 may be formed preliminarily on either one of the monocrystalline compound semiconductor layer or the Si supporting substrate 34, or the three sheets may be bonded with interposition of an insulative thin film.

Subsequently, the substrates are separated at the porous Si layer 32 as shown in Fig. 3D. On the Si supporting substrate, the layers have the structure of porous Si 32 / monocrystalline compound semiconductor layer 33 / insulative layer 35 / Si supporting substrate 34.

The porous Si 32 is removed selectively by chemical etching by use of an etching solution which is capable of etching Si at a higher etching rate than the compound semiconductor to leave the thin-layered monocrystalline compound semiconductor layer 33 on the insulative substrate 35 and 34.

Otherwise, the porous Si 32 is selectively removed by grinding by utilizing the monocrystalline compound semiconductor layer 33 as the grinding stopper.

Fig. 3E illustrates a semiconductor substrate of the present invention. The monocrystalline compound semiconductor layer 33 is formed flat and uniformly in a thin layer on the insulative substrate 35 and 34 over the entire large area of the wafer. The resulting semiconductor substrate is useful as a compound semiconductor substrate and for production of insulation-isolated electronic elements.

When the substrate is used as a compound semiconductor substrate, the insulative layer 35 is not essential.

The first monocrystalline Si substrate 31 may be repeatedly used for the same use after removal of the remaining Si and surface flattening treatment if the surface has become roughened unacceptably in the next production cycle.

Embodiment 4

A first monocrystalline Si substrate 41 is made porous at the surface to form a porous layer 42 as shown in Fig. 4A. Then a nonporous monocrystalline compound semiconductor layer 43 is formed on the porous Si layer 42 as shown in Fig. 4B.

A light-transmissive supporting substrate 44 is brought into close contact with the monocrystalline compound semiconductor layer 43 with interposition of an insulative layer 45 at room temperature as shown in Fig. 4C, and then the contacted matter was subjected to anode coupling, compression, heat treatment, or combination of the treatments to bond tightly the light-transmissive supporting substrate 44 with the monocrystalline layer 43 with interposition of the insulative layer 45. The insulative layer 45 may be formed preliminarily on either one of the monocrystalline compound semiconductor layer or the light-transmissive supporting substrate 44, or the three sheets may be bonded with interposition of an insulative thin film.

Subsequently, the substrates are separated at the porous Si layer 42 as shown in Fig. 4D. On the light-transmissive supporting substrate, the layers have the structure of porous Si 42 / monocrystalline compound semiconductor layer 43 / insulative layer 45 / light-transmissive supporting substrate 44.

The porous Si 42 is removed selectively by chemical etching by use of an etching solution which is capable of etching Si at a higher etching rate than the compound semiconductor to leave a thin-layered monocrystalline compound semiconductor layer 43 on the insulative substrate 45 and 44.

Otherwise, the porous Si 42 is selectively removed by grinding by utilizing the monocrystalline compound semiconductor layer 43 as the grinding stopper.

Fig. 4E illustrates a semiconductor substrate of the present invention. The monocrystalline compound semiconductor layer 43 is formed flat and uniformly in

a thin layer on the insulative substrate 45 and 44 over
the entire large area of the wafer. The resulting
semiconductor substrate is useful for production of
insulation-isolated electronic elements.

The insulative layer 45 is not essential in this
embodiment.

The first monocrystalline Si substrate 41 may be
repeatedly used for the same use after removal of the
remaining Si and surface flattening treatment if the
surface has become roughened unacceptably in the next
production cycle.

Embodiment 5

A first monocrystalline Si substrate 51 is made porous
at the both faces to form porous layers 52, 53 as shown
in Fig. 5A. Then, nonporous monocrystalline compound
semiconductor layers 54, 55 are formed on the porous Si
layers 52, 53 as shown in Fig. 5B.

Two supporting substrates 56, 57 are brought into close
contact with the monocrystalline semiconductor layers
54, 55 with interposition of insulative layers 58, 59
respectively at room temperature as shown in Fig. 5C,
and then the contacted matter is subjected to anode
coupling, compression, heat treatment, or combination
of the treatments to bond tightly the supporting
substrates 56, 57 and the monocrystalline layers 54, 55
with interposition of the insulative layers 58, 59. In
the bonding, the respective insulative layers 58, 59
may be formed preliminarily on either one of the
monocrystalline semiconductor layer 54, 55 or the
supporting substrate 56, or the five sheets may be
bonded with interposition of insulative thin films.

Subsequently, the substrates are separated into three at the both porous Si layers 52, 53 as shown in Fig. 5D. The two supporting substrates come to have a structure of porous Si / monocrystalline semiconductor layer / insulative layer / supporting substrate (52/54/58/56, and 53/55/59/57).

The porous Si layers 52, 53 are removed selectively by chemical etching to leave thin-layered monocrystalline semiconductor layers 54, 55 on the supporting substrates 58/56 and 59/57.

Otherwise, the porous Si 52, 53 is selectively removed by grinding by utilizing the monocrystalline semiconductor layers 54, 55 as the grinding stopper.

Fig. 5E illustrates semiconductor substrates prepared according to the present invention. The monocrystalline compound semiconductor layers are formed flat and uniformly in a thin layer on the supporting substrates over the entire large area of the two wafers at a time with a large area. The resulting semiconductor substrate is useful for production of insulation-isolated electronic elements.

The insulative intervening layers 58, 59 are not essential.

The supporting substrates 56, 57 need not be the same.

The first monocrystalline Si substrate 51 may be repeatedly used for the same use after removal of the remaining Si and surface flattening treatment if the surface has become roughened unacceptably in the next production cycle.

Example 1

A first monocrystalline (100) Si substrate of P-type
having a diameter of 6 inches, a thickness of 625 μm,
and a specific resistance of 0.01 Ω.cm was anodized in
an HF solution under the anodization conditions as
below:

Current density:	5 mA.cm^{-2}
Anodization solution:	HF:H$_2$O:C$_2$H$_5$OH = 1:1:1
Time:	12 minutes
Thickness of porous Si:	10 μm
Porosity:	15 %

This substrate was oxidized at 400°C in an oxygen
atmosphere for one hour. Thereby the inner wall of the
pores of the porous Si was covered with a thermal
oxidation film. On the porous Si, monocrystalline Si
was allowed to grow epitaxially in a thickness of 1 μm
by CVD (chemical vapor deposition) under the growth
conditions below:

Source gas:	SiH$_2$Cl$_2$/H$_2$
Gas flow rate:	0.5/180 ℓ/min
Gas pressure:	80 Torr
Temperature:	950°C
Growth rate:	0.3 μm/min

The face of the epitaxially grown Si layer was
thermally oxidized to form an SiO$_2$ layer of 100 nm
thick.

On the face of this Si substrate, a separately prepared
second Si substrate having an SiO$_2$ layer of 500 nm thick
was superposed with the SiO$_2$ layer inside, and the
superposed matter was heat-treated at 900°C for 2 hours
to bond the substrates tightly.

A pulling force was applied to the resulting bonded
wafer in the direction perpendicular to the wafer face
in such a manner that a plate was bonded respectively

to each of the both faces of the wafer with an adhesive and the plates were pulled to opposite directions with a jig. Consequently, the porous Si layer was broken to cause separation of the wafer into two sheets with the porous Si layers exposed.

The porous Si layer on the second substrate was etched selectively in a mixture of 49% hydrofluoric acid and 30% hydrogen peroxide (1:5) with agitation. The porous Si was etched and removed completely with the monocrystalline Si remaining unetched as an etching stopper. The etching rate of the nonporous monocrystalline Si was extremely low, the selection ratio of the etching rate of the porous Si being 105 or higher. Therefore, the decrease in thickness of the nonporous layer by etching was practicably negligible (several tens of Å).

Consequently, a monocrystalline Si layer was formed in a thickness of 1 μm on an Si oxide film. The monocrystalline Si layer did not change at all by the selective etching of the porous Si layer.

The cross-section of the Si layer was observed by transmission electron microscopy, and it was confirmed that no additional crystal defect was formed in the Si layer and the excellent crystallinity was retained.

Thus an SOI substrate was obtained which has a semiconductor layer of high quality.

The other Si substrate having been separated at the porous Si layer portion was etched in the same manner as above to remove the remaining porous layer, and its surface was polished. The obtained Si substrate was used repeatedly for the same use in the next production

cycle. Thereby a plurality of SOI substrates having a
semiconductor layer of high quality were obtained.

Example 2

A first monocrystalline (100) Si substrate of P-type
having a diameter of 4 inches, a thickness of 525 μm,
and a specific resistance of 0.01 Ω.cm was anodized in
an HF solution under the anodization conditions as
below:

Current density:	7 mA. cm^{-2}
Anodization solution:	HF:H$_2$O:C$_2$H$_5$OH = 1:1:1
Time:	12 minutes
Thickness of porous Si:	10 μm
Porosity:	15 %

This substrate was oxidized at 400°C in an oxygen
atmosphere for 2 hours. Thereby the inner wall of the
pores of the porous Si was covered with a thermal
oxidation film. On the porous Si, monocrystalline Si
was allowed to grow epitaxially in a thickness of 0.5
μm by MBE (molecular beam epitaxy) under the growth
conditions below:

Temperature:	700°C
Pressure:	1 x 10^{-9} Torr
Growth rate:	0.1 μm/sec
Temperature:	950°C
Growth rate:	0.3 μm/min

The face of the epitaxially grown Si layer was
thermally oxidized to form an SiO$_2$ layer of 100 nm
thick.

On the face of the SiO$_2$ layer, was superposed a
separately prepared fused quartz substrate, and the
superposed matter was heat-treated at 400°C for 2 hours
to bond the substrates.

A sufficient compression force was applied uniformly to the resulting bonded wafer in the direction perpendicular to the wafer face such that plates were bonded to each of the both faces of the wafer with an adhesive and the compression force was applied with the same jig as in Example 1.

Consequently, the porous Si layer was broken to cause separation of the wafer into two sheets with the porous Si layers exposed.

The porous Si layers were etched selectively in a mixture of buffered hydrofluoric acid and 30% hydrogen peroxide (1:5) with agitation. Thereby the porous Si was etched and removed completely with the monocrystalline Si remaining unetched as an etch-stop material. The etching rate of the nonporous monocrystalline Si was extremely low, the selection ratio of the etching rate of the porous Si being, 105 or higher. Therefore, the decrease in thickness of the nonporous layer by etching was practicably negligible (several tens of Å).

Consequently, a monocrystalline Si layer was formed in a thickness of 0.5 μm on a fused quartz substrate. The monocrystalline Si layer did not change at all by the selective etching of the porous Si layer.

The cross-section of the Si layer was observed by transmission electron microscopy, and it was confirmed that no additional crystal defect was formed in the Si layer and the excellent crystallinity was retained.

A plurality of SOI substrates having a semiconductor layer of high quality were prepared by repeating the above process in the same manner as in Example 1.

Example 3

A first monocrystalline (100) Si substrate of P-type or
N-type having a diameter of 6 inches, a thickness of
625 μm, and a specific resistance of 0.01 Q.cm was
anodized in an HF solution under the anodization
conditions as below:

      Current density: 7 mA.cm^{-2}
      Anodization solution: HF:H$_2$O:C$_2$H$_5$OH = 1:1:1
      Time: 12 minutes
      Thickness of porous Si: 10 μm
      Porosity: 15 %

This substrate was oxidized at 400°C in an oxygen
atmosphere for one hour. Thereby the inner wall of the
pores of the porous Si was covered with a thermal
oxidation film. On the porous Si, monocrystalline GaAs
was allowed to grow epitaxially in a thickness of 1 μm
by MOCVD (metal organic chemical vapor deposition)
under the growth conditions below:

      Source gas: TMG / AsH$_3$ / H$_2$
      Gas pressure: 80 Torr
      Temperature: 700°C

On the face of the formed GaAs layer, was superposed a
separately prepared second Si substrate, and the
superposed matter was heat-treated at 900°C for one
hour to bond the substrates tightly.

A sufficient compression force was applied to the
resulting bonded wafer in the same manner as in Example
2. Thereby, the porous Si layer was broken to allow
the wafer to separate into two sheets with the porous
Si layers exposed.

Then, the oxide film on the inner wall of the porous Si
layer was removed by hydrofluoric acid, and the porous

Si was etched with a mixture of ethylene diamine, pyrocathecol, and water (17 ml : 3 g : 8 ml) at 110°C. Thereby the porous Si was etched selectively and removed completely with the monocrystalline GaAs remaining unetched as an etch-stopping material. The etching rate of the nonporous monocrystalline GaAs was extremely low and practicably negligible.

Consequently, a monocrystalline GaAs layer was formed in a thickness of 1 μm on a Si substrate. The monocrystalline GaAs layer did not change at all by the selective etching of the porous Si layer.

The cross-section of the GaAs layer was observed by transmission electron microscopy, and it was confirmed that no additional crystal defect was formed in the GaAs layer and the excellent crystallinity was retained.

A plurality of semiconductor substrates having a GaAs layer of high quality were prepared by repeating the above process in the same manner as in Example 2.

GaAs on an insulative film was also prepared by employing an Si substrate having an oxide film as the supporting substrate.

Example 4

A first monocrystalline (100) Si substrate of P-type or N-type having a diameter of 5 inches, a thickness of 625 ,um, and a specific resistance of 0.01 n. cm was anodized in an HF solution under the anodization conditions as below:

Current density: 10 mA.cm$_{-2}$

Anodization solution: $HF:H_2O:C_2H_5OH = 1:1:1$
Time: 24 minutes
Thickness of porous Si: 20 μm
Porosity: 17 %

This substrate was oxidized at 400°C in an oxygen atmosphere for 2 hours. Thereby the inner wall of the pores of the porous Si was covered with a thermal oxidation film. On the porous Si, monocrystalline AlGaAs was allowed to grow epitaxially in a thickness of 0.5 μm by MBE (molecular beam epitaxy).

On the face of the formed AlGaAs layer, was superposed a face of a separately prepared low-melting glass substrate. The superposed matter was heat-treated at 500°C for 2 hours to bond the substrates tightly.

A sufficient compression force was applied to the resulting bonded wafer in the same manner as in Example 2. Thereby, the porous Si layer was broken to allow the wafer to separate into two sheets with the porous Si layers exposed.

The porous Si was etched with hydrofluoric acid solution. Thereby the porous Si was etched selectively and removed off completely with the monocrystalline AlGaAs remaining unetched as an etch-stopping material. The etching rate of the nonporous monocrystalline AlGaAs was extremely low and practicably negligible.

Consequently, a monocrystalline AlGaAs layer was formed in a thickness of 0.5 ~m on a glass substrate. The monocrystalline AlGaAs layer did not change at all by the selective etching of the porous Si layer.

The cross-section of the AlGaAs layer was observed by transmission electron microscopy, and it was confirmed

that no additional crystal defect was formed in the
AlGaAs layer and the excellent crystallinity was
retained.

A plurality of semiconductor substrates having a GaAs
layer of high quality were prepared by repeating the
above process in the same manner as in Example 2.

Example 5

A first monocrystalline (100) Si substrate of P-type or
N-type having been polished on the both faces and
having a diameter of 6 inches, a thickness of 625 μm,
and a specific resistance of 0.01 Ω.cm was anodized on
the both faces in an HF solution under the anodization
conditions below:

Current density:	5 mA.cm^{-2}
Anodization solution:	HF:H$_2$O:C$_2$H$_5$OH = 1:1:1
Time:	12 x 2 minutes
Thickness of porous Si:	10 μm each
Porosity:	15 %

This substrate was oxidized at 400°C in an oxygen
atmosphere for one hour. Thereby the inner wall of the
pores of the porous Si was covered with a thermal
oxidation film. On the porous Si formed on the both
faces of the substrate, monocrystalline Si was allowed
to grow epitaxially in a thickness of 1 μm respectively
by CVD (chemical vapor deposition) under the growth
conditions below:

Source gas:	SiH$_2$Cl$_2$/H$_2$
Gas flow rate:	0.5/180 ℓ/min
Gas pressure:	80 Torr
Temperature:	950°C
Growth rate:	0.3 μm/min

The faces of the formed epitaxial Si layers were thermally oxidized to form SiO_2 layers in a thickness of 100 nm.

On each of the faces of the SiO_2 layers, a separately prepared Si substrate having a 500-nm thick SiO_2 layer was superposed respectively with the SiO_2 layers inside, and the superposed matter was heat-treated at 600°C for 2 hours to bond the substrates tightly.

A sufficient pulling force was applied to the resulting bonded wafer in the direction perpendicular to the bonded wafer face in the same manner as in Example 1. Thereby, the two porous Si layers were broken to allow the wafer to separate into three sheets with the porous Si layers exposed.

The porous Si layers were etched selectively with a mixture of 49% hydrofluoric acid with 30% hydrogen peroxide (1:5) with agitation. Thereby the porous Si was etched selectively and removed completely with the monocrystalline Si remaining unetched as an etch-stopping material. The etching rate of the nonporous monocrystalline Si was extremely low, the selection ratio of the etching rate of the porous Si being 105 or higher. Therefore, the decrease in thickness of the nonporous layer by etching was practicably negligible (several tens of Å).

Consequently, a monocrystalline Si layer was formed in a thickness of 1 μm respectively on the two Si oxide films simultaneously. The monocrystalline Si layers did not change at all by the selective etching of the porous Si layer.

The cross-section of the Si layer was observed by transmission electron microscopy, and it was confirmed

that no additional crystal defect was formed in the Si layer and the excellent crystallinity was retained.

A plurality of semiconductor substrates having a semiconductor layer of high quality were prepared by repeating the above process in the same manner as in Example 1.

Example 6

A first monocrystalline (100) Si substrate of P-type or N-type having a diameter of 5 inches, a thickness of 625 ~m, and a specific resistance of 0.01 Ω.cm was anodized in an HF solution under the anodization conditions below:

Current density: 7 mA.cm$_{-2}$
Anodization solution: HF:H_2O:C_2H_5OH = 1:1:1
Time: 4 minutes
Thickness of porous Si: 3 μm
Porosity: 15 %

The anodization was conducted further under the conditions below:

Current density: 30 mA.cm^{-2}
Anodization solution: HF:H_2O:C_2H_5OH = 1:3:2
Time: 3 minutes
Thickness of porous Si: 10 μm
Porosity: 45 %

This substrate was oxidized at 400°C in an oxygen atmosphere for one hour. Thereby the inner wall of the pores of the porous Si was covered with a thermal oxidation film. On the porous Si formed on the substrate, monocrystalline Si was allowed to grow epitaxially in a thickness of 0.3 μm by CVD under the conditions below:

Source gas: SiH_4

Carrier gas: H_2
Temperature: 850°C
Pressure: 1×10^{-2} Torr
Growth rate: 3.3 nm/sec

The surface of the formed epitaxial Si layer was
thermally oxidized to form SiO_2 layer in a thickness of
100 nm.
On the face of the SiO_2 layer, a separately prepared
second Si substrate having a 500-nm thick SiO_2 layer was
superposed with the SiO_2 layer inside, and the
superposed matter was heat-treated at 700°C for 2 hours
to bond the substrates tightly.

A sufficient pulling force was applied to the resulting
bonded wafer in the direction perpendicular to the
bonded wafer face in the same manner as in Example 1.
Thereby, the porous Si layer was broken to allow the
wafer to separate into two sheets with the porous Si
layers exposed.

The porous Si on the second Si substrate was etched
selectively with an etching solution of $HF/HNO_2/CH_3COOH$
type. Thereby the porous Si was etched selectively and
removed completely. The etching rate of the nonporous
monocrystalline Si was extremely low, so that the
thickness decrease of the nonporous layer by etching
was practicably negligible.

Consequently, a monocrystalline Si layer was formed in
a thickness of 1 μm on the Si oxide film. The
monocrystalline Si layers did not change at all by the
selective etching of the porous Si layer.

The cross-section of the Si layer was observed by
transmission electron microscopy, and it was confirmed

that no additional crystal defect was formed in the Si
layer and the excellent crystallinity was retained.
A plurality of semiconductor substrates having a
semiconductor layer of high quality were prepared by
repeating the above process in the same manner as in
Example 1.

Example 7

A first monocrystalline (100) Si substrate of P-type or
N-type having a diameter of 6 inches, a thickness of
625 am, and a specific resistance of 0.01 Q.cm was
anodized in an HF solution under the anodization
conditions below:

Current density: 5 mA.cm^{-2}

Anodization solution: $HF:H_2O:C_5H_5OH = 1:1:1$

Time: 12 minutes

Thickness of porous Si: 10 μm

Porosity: 15 %

This substrate was oxidized at 400°C in an oxygen
atmosphere for one hour. Thereby the inner wall of the
pores of the porous Si was covered with a thermal
oxidation film. On the porous Si formed on the
substrate, monocrystalline Si was allowed to grow
epitaxially in a thickness of 1 gm by CVD under the
growth conditions below:

Source gas: SiH_2Cl_2/H_2

Gas flow rate: 0.5/180 ℓ/min

Gas pressure: 80 Torr

Temperature: 950°C

Growth rate: 0.3 μm/min

The surface of the formed epitaxial Si layer was
thermally oxidized to form SiO$_2$ layer in a thickness of
100 nm.

On the face of the SiO$_2$ layer, a separately prepared
second Si substrate having a 500-nm thick SiO$_2$ layer was
superposed with the SiO$_2$ layer inside, and the
superposed matter was heat-treated at 900°C for 2 hours
to bond the substrates tightly.

A sufficient pulling force was applied to the resulting
bonded wafer in the direction perpendicular to the
bonded wafer face in the same manner as in Example 1.
Thereby, the porous Si layer was broken to allow the
wafer to separate into two sheets with the porous Si
layers exposed.

The porous Si layer on the second substrate was ground
selectively by utilizing the monocrystalline layer as
the stopper. Thereby the porous Si was removed
selectively.

Consequently, a monocrystalline Si layer was formed in
a thickness of 1 ,um on the Si oxide film. The
monocrystalline Si layers did not change at all by the
selective grinding of the porous Si layer.

The cross-section of the Si layer was observed by
transmission electron microscopy, and it was confirmed
that no additional crystal defect was formed in the Si
layer and the excellent crystallinity was retained.

A plurality of semiconductor substrates having a
semiconductor layer of high quality were prepared by
repeating the above process in the same manner as in
Example 1.

Example 8

A first monocrystalline (100) Si substrate of P-type or
N-type having a diameter of 6 inches, a thickness of

625 μm and a specific resistance of 0.01 Ω.cm was anodized in an HF solution under the anodization conditions below:

Current density: 5 mA.cm^{-2}

Anodization solution: HF:H$_2$O:C$_2$H$_5$OH = 1:1:1

Time: 12 minutes

Thickness of porous Si: 10 μm

Porosity: 15 %

This substrate was oxidized at 400°C in an oxygen atmosphere for one hour. Thereby the inner wall of the pores of the porous Si was covered with a thermal oxidation film. On the porous Si formed on the substrate, monocrystalline Si was allowed to grow epitaxially in a thickness of 1 μm by CVD under the conditions below:

Source gas: SiH$_2$Cl$_2$/H$_2$

Gas flow rate: 0.5/180 ℓ/min

Gas pressure: 80 Torr

Temperature: 950°C

Growth rate: 0.3 μm/min

The surface of the formed epitaxial Si layer was thermally oxidized to form SiO$_2$ layer in a thickness of 100 nm.

On the face of the SiO$_2$ layer, a separately prepared second Si substrate having a 500-nm thick SiO$_2$ layer was superposed with the SiO$_2$ layer inside, and the superposed matter was heat-treated at 900°C for 2 hours to bond the substrates tightly.

A supersonic energy was applied to the resulting bonded wafer in a vessel provided with a supersonic oscillator. Thereby, the porous Si layer was broken to allow the wafer to separate into two sheets with the porous Si layers exposed.

The porous Si layer on the second Si substrate was
etched selectively with a mixture of 49% hydrofluoric
acid with 30% hydrogen peroxide (1:5) with agitation.
Thereby the porous Si was etched selectively and
removed completely with the monocrystalline Si
remaining unetched as an etch-stopping material. The
etching rate of the nonporous monocrystalline Si was
extremely low, the selection ratio of the etching rate
of the porous Si being 105 or higher. Therefore, the
decrease in thickness of the nonporous layer by etching
was practicably negligible (several tens of Å).

Consequently, a monocrystalline Si layer was formed in
a thickness of 1 ,um on the Si oxide film. The
monocrystalline Si layers did not change at all by the
selective etching of the porous Si layer.

The cross-section of the Si layer was observed by
transmission electron microscopy, and it was confirmed
that no additional crystal defect was formed in the Si
layer and the excellent crystallinity was retained.

The first monocrystalline Si substrate was used
repeatedly for the same use after removal of the porous
Si remaining thereon.

Example 9

A first monocrystalline (100) Si substrate of P-type or
N-type having a diameter of 4 inches, a thickness of
525 μm, and a specific resistance of 0.01 Ω.cm was
anodized in an HF solution under the anodization
conditions as below:

    Current density: 7 mA.cm^{-2}
    Anodization solution: HF:H$_2$O:C$_2$H$_5$OH = 1:1:1
    Time: 12 minutes
    Thickness of porous Si: 10 μm

Porosity: 15 %

This substrate was oxidized at 400°C in an oxygen atmosphere for 2 hours. Thereby the inner wall of the pores of the porous Si was covered with a thermal oxidation film. On the porous Si, monocrystalline Si was allowed to grow epitaxially in a thickness of 0.5 μm by MBE (molecular beam epitaxy) under the growth conditions below:

Temperature: 700°C
Pressure: 1 x 10^{-9} Torr
Growth rate: 0.1 nm/sec
Temperature: 950°C
Growth rate: 0.3 μm/min

The surface of the epitaxially grown Si layer was thermally oxidized to form an SiO$_2$ layer of 100 nm thick.

On the face of the SiO$_2$ layer, was superposed a separately prepared fused quartz substrate, and the superposed matter was heat-treated at 400°C for 2 hours to bond the substrates.

The end of the porous layer was bared to the edge face of the wafer, and the porous Si is slightly etched. Thereto, a sharp blade like a shaver blade was inserted. Thereby, the porous layer was broken, and the wafer was separated into two sheets with the porous Si layers exposed.

The porous Si layer on the fused quartz substrate was etched selectively in a mixture of buffered hydrofluoric acid and 30% hydrogen peroxide (1:5) with agitation. Thereby the porous Si was etched and removed completely with the monocrystalline Si

remaining unetched as an etch-stopping material. The etching rate of the nonporous monocrystalline Si was extremely low, the selection ratio of the etching rate of the porous Si being, 105 or higher. Therefore, the decrease in thickness of the nonporous layer by etching was practicably negligible (several tens of Å).

Consequently, a monocrystalline Si layer was formed in a thickness of 0.5 ,um on a fused quartz substrate. The monocrystalline Si layer did not change at all by the selective etching of the porous Si layer.

The cross-section of the Si layer was observed by transmission electron microscopy, and it was confirmed that no additional crystal defect was formed in the Si layer and the excellent crystallinity was retained.

The same results were obtained without forming the oxide film of the surface of the epitaxial Si surface. The first monocrystalline Si substrate was used repeatedly for the same use after removal of the remaining porous Si and mirror-polishing of the surface.

Example 10

A first monocrystalline (100) Si substrate of P-type or N-type having a polished face on each side and having a diameter of 6 inches, a thickness of 625 μm, and a specific resistance of 0.01 Ω.cm was anodized on both sides in an HF solution under the anodization conditions below:

        Current density: 5 mA.cm^{-2}
        Anodization solution: HF:H$_2$O:C$_2$H$_5$OH = 1:1:1
        Time: 12 x 2 minutes
        Thickness of porous Si: 10 μum each
        Porosity: 15 %

This substrate was oxidized at 400°C in an oxygen atmosphere for one hour. Thereby the inner wall of the pores of the porous Si was covered with a thermal oxidation film. On the porous Si formed on both faces of the substrate, monocrystalline Si was allowed to grow epitaxially in a thickness of 1 μm by CVD (chemical vapor deposition) under the conditions below:

Source gas:	SiH_2Cl_2/H_2
Gas flow rate:	0.5/180 ℓ/min
Gas pressure:	80 Torr
Temperature:	950°C
Growth rate:	0.3 μm/min

The surfaces of the formed epitaxial Si layers were thermally oxidized to form SiO_2 layers in a thickness of 100 nm.

On each of the faces of the SiO_2 layers, a separately prepared second Si substrate having a 500-nm thick SiO_2 layer was superposed with the SiO_2 layer inside, and the superposed matter was heat-treated at 600°C for 2 hours to bond the substrates tightly.

The porous layers were bared at the edge face of the wafer, and a liquid such as water was allowed to penetrate into the porous Si. The entire bonded wafer was heated or cooled, whereby the porous Si layers were broken owing to expansion or other causes to allow the wafer to separate into three sheets with the porous Si layers exposed.

The porous Si layers were etched selectively with a mixture of 49% hydrofluoric acid with 30% hydrogen peroxide (1:5) with agitation. Thereby the porous Si was etched selectively and removed completely with the monocrystalline Si remaining unetched as an etch-stopping material. The etching rate of the

nonporous monocrystalline Si was extremely low, the selection ratio of the etching rate of the porous Si being 105 or higher. Therefore, the decrease in thickness of the nonporous layer by etching was practicably negligible (several tens of Å).

Consequently, a monocrystalline Si layer was formed in a thickness of 1 μm respectively on the two Si oxide films simultaneously. The monocrystalline Si layers did not change at all by the selective etching of the porous Si layer.

The cross-section of the Si layer was observed by transmission electron microscopy, and it was confirmed that no additional crystal defect was formed in the Si layer and the excellent crystallinity was retained.

The same results were obtained without formation of the oxide film on the surface of the epitaxial Si layer.

The first monocrystalline Si substrate was used repeatedly for the same use after removal of the remaining porous Si and flattening of the surface by hydrogen treatment.

Example 11

A first monocrystalline (100) Si substrate of P-type or N-type having a diameter of 5 inches, a thickness of 625 ,um, and a specific resistance of 0.01 Q.cm was anodized in an HF solution under the anodization conditions below:

Current density:	7 mA.cm^{-2}
Anodization solution:	$HF:H_2O:C_2H_5OH = 1:1:1$
Time:	4 minutes
Thickness of porous Si:	3 μm
Porosity:	15 %

The anodization was conducted further under the
conditions below:

    Current density: 30 mA.cm^{-2}

    Anodization solution: HF:H$_2$O:C$_2$H$_5$OH = 1:3:2

    Time: 3 minutes

    Thickness of porous Si: 10 μm

    Porosity: 45 %

This substrate was oxidized at 400°C in an oxygen
atmosphere for one hour. Thereby the inner wall of the
pores of the porous Si was covered with a thermal
oxidation film. On the porous Si formed on the
substrate, monocrystalline Si was allowed to grow
epitaxially in a thickness of 0.3 μm by CVD under the
conditions below:

    Source gas: SiH$_4$

    Carrier gas: H$_2$

    Temperature: 850°C

    Pressure: 1 x 10^{-2} Torr

    Growth rate: 3.3 nm/sec

The surface of the formed epitaxial Si layer was
thermally oxidized to form a SiO$_2$ layer in a thickness
of 100 nm.

On the face of the SiO$_2$ layer, a separately prepared
second Si substrate having a 500-nm thick SiO$_2$ layer was
superposed with the SiO$_2$ layer inside, and the
superposed matter was heat-treated at 700°C for 2 hours
to bond the substrates tightly.

A force was applied to the first (or second) substrate
in a direction parallel to the second (or first)
substrate, whereby the porous Si layer was broken by
the shear stress to allow the wafer to separate into
two sheets with the porous Si layers exposed.

The porous Si layer was etched selectively with an HF/HNO$_3$/CH$_3$COOH type etching solution. Thereby the porous Si was etched selectively and removed completely. The etching rate of the nonporous monocrystalline Si was extremely low, so that the decrease in thickness of the nonporous layer by etching was practicably negligible.

Consequently, a monocrystalline Si layer was formed in a thickness of 1 μm on the Si oxide layer. The monocrystalline Si layer did not change at all by the selective etching of the porous Si layer.

The cross-section of the Si layer was observed by transmission electron microscopy, and it was confirmed that no additional crystal defect was formed in the Si layer and the excellent crystallinity was retained.

The same results were obtained without forming the oxide film on the surface of the epitaxial Si layer surface.

The first monocrystalline Si substrate was used repeatedly for the same use after removal of the remaining porous Si.

Example 12

A first monocrystalline (100) Si substrate of P-type or N-type having a diameter of 5 inches, a thickness of 625 ,um, and a specific resistance of 0.01 Q.cm was anodized in an HF solution under the anodization conditions below:

Current density:	7 mA.cm^{-2}
Anodization solution:	HF:H$_2$O:C$_2$H$_5$OH = 1:1:1
Time:	4 minutes
Thickness of porous Si:	3 μm

Porosity: 15 %

The anodization was conducted further under the conditions below:

Current density: 30 mA.cm^{-2}

Anodization solution: HF:H_2O:C_2H_5OH = 1:3:2

Time: 3 minutes

Thickness of porous Si: 10 μm

Porosity: 45 %

This substrate was oxidized at 400°C in an oxygen atmosphere for one hour. Thereby the inner wall of the pores of the porous Si was covered with a thermal oxidation film. On the porous Si formed on the substrate, monocrystalline Si was allowed to grow epitaxially in a thickness of 0.3 μm by CVD under the conditions below:

Source gas: SiH_4

Carrier gas: H_2

Temperature: 850°C

Pressure: 1 x 10^{-2} Torr

Growth rate: 3.3 nm/sec

The surface of the formed epitaxial Si layer was thermally oxidized to form a SiO_2 layer in a thickness of 100 nm.

On the face of the SiO_2 layer, a separately prepared second Si substrate having a 500-nm thick SiO_2 layer was superposed with the SiO_2 layer inside, and the superposed matter was heat-treated at 700°C for 2 hours to bond the substrates tightly.

The porous layers were bared at the edge face of the wafer, and the porous Si was etched from the edge face with a selective etching solution, whereby the wafer came to be separated into two sheets.

Further, the porous Si layer on the second Si substrate was etched selectively with an $HF/HNO_3/CH_3COOH$ type etching solution. Thereby the porous Si was etched selectively and removed completely. The etching rate of the nonporous monocrystalline Si was extremely low, so that the thickness decrease of the nonporous layer by etching was practicably negligible.

Consequently, a monocrystalline Si layer was formed in a thickness of 1 μm on the Si oxide film. The monocrystalline Si layers did not change at all by the selective etching of the porous Si layer.

The cross-section of the Si layer was observed by transmission electron microscopy, and it was confirmed that no additional crystal defect was formed in the Si layer and the excellent crystallinity was retained.

The same results were obtained without forming the oxide film on the surface of the epitaxial Si layer surface.

The first monocrystalline Si substrate was used repeatedly for the same use after removal of the remaining porous Si.

ABSTRACT OF THE DISCLOSURE

A process for producing a semiconductor substrate is provided which comprises steps of forming a porous layer on a first substrate, forming a nonporous monocrystalline semiconductor layer on the porous layer of the first substrate, bonding the nonporous monocrystalline layer onto a second substrate, separating the bonded substrates at the porous layer, removing the porous layer on the second substrate, and removing the porous layer constituting the first substrate.

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PROCESS FOR PRODUCTION OF SEMICONDUCTOR SUBSTRATE

BACKGROUND OF THE INVENTION

Field of the Invention

5   The present invention relates to a process for producing a semiconductor substrate. More specifically, the present invention relates to a process for producing a monocrystalline semiconductor on a dielectric-isolated or insulative material, or a

10   monocrystalline compound semiconductor on a semiconductor substrate. Further the present invention relates to a process for producing an electronic device or an integrated circuit formed on a single crystalline semiconductor layer.

Related Background Art

The technique of formation of monocrystalline Si (silicon) semiconductor on an insulative material is well known as silicon-on-insulator (SOI) technique. The device prepared by SOI technique has various

20   advantages which are not achievable by a bulk Si substrate of usual Si integrated circuits as below:

1. Ease of dielectric isolation, and possibility of high degree of integration,

2. High resistance against radioactive rays,

25   3. Low floating capacity, and possibility of high speed operation,

4. Needlessness of the welling process,

5. Preventability of latch-up,

6. Possibility of producing a complete depletion type field-effect transistor,

and so forth.

5   The process of forming the SOI structure has been actively studied for several decades. The results of the studies are summarized, for example, in the paper: Special Issue; "Single-crystal silicon on non-single-crystal insulators"; edited by G.W. Cullen,

10   Journal of Crystal Growth; Vol.63, No.3, pp.429-590 (1983).

SOS (silicon on sapphire) is known which is produced by heteroepitaxial growth of silicon on monocrystalline sapphire by CVD (chemical vapor

15   deposition). The SOS technique, which is successful as one of the SOI techniques, is limited in the application fields, because of many crystal defects caused by mismatch of the lattice at the interface between the Si layer and the underlying sapphire,

20   contamination of the Si layer with aluminum from the sapphire substrate, expensiveness of the substrate, and difficulty of large-area substrate formation.

Recently, studies are being made to produce the SOI structure without using a sapphire substrate. The

25   studies are classified roughly into the two processes below:

1. A first process including surface oxidation of a

monocrystalline Si substrate, local exposure of the Si

substrate by opening a window, and epitaxial growth of

Si laterally from the exposed portion as the seed to

form an Si layer on SiO_2. (Si layer deposition on SiO_2)

5   2. A second process including SiO_2 formation beneath a

monocrystalline SiO_2 substrate, utilizing the SiO_2

substrate as the active layer. (No Si layer deposition)

The device formed on a compound semiconductor

exhibits performances, such as high speed, and

10   luminescence, which are not achievable by Si. Such

types of devices are formed by epitaxial growth on a

compound semiconductor substrate such as GaAs. The

compound semiconductor substrate, however, has

disadvantages of high cost, low mechanical strength,

15   and difficulty in formation of a large-area wafer.

Accordingly, heteroepitaxial growth of a compound

semiconductor on an Si wafer is being studied to attain

low cost, high mechanical strength, and ease of

production of a large-area wafer.

20   The above known process of the item 1 (Si layer

deposition on SiO_2) includes methods of direct lateral

epitaxial growth of monocrystalline Si layer by CVD;

deposition of amorphous Si and subsequent heat

treatment to cause solid-phase lateral epitaxial

25   growth; melting recrystallization to grow

monocrystalline layer on an SiO_2 by irradiation of

amorphous or polycrystalline Si layer with focused

energy beam such as electron beam and laser beam; and
zone melting recrystallization in which a bar-shaped
heater is moved to scan with a belt-like melt zone.
These methods respectively have advantages and

5   disadvantages, involving problems in process
controllability, productivity, product uniformity, and
product quality, and are not industrialized yet. For
example, the CVD method requires sacrificial oxidation,
giving low crystallinity in the solid-phase growth.

10   The beam annealing method involves problems in
processing time of focused beam scanning and in
controllability of beam superposition and focusing. Of
the above methods, the zone melting recrystallization
is the most advanced method, and is employed in

15   relatively large scale integrated circuits. This
method, however, still causes crystal defects in sub-
grain boundaries, not being successful in production of
a minority carrier device.

The above known process of the item 2 in which

20   the Si substrate is not utilized as the seed for
epitaxial growth includes the four methods below:

1. An oxidation film is formed on a
monocrystalline Si substrate which has V-shaped grooves
on the surface formed by anisotropical etching; a

25   polycrystalline Si layer is deposited in a thickness
approximate to that of the Si substrate on the
oxidation film; and the back face of the Si substrate

is ground to form a monocrystalline Si region isolated
dielectrically by surrounding with the V-shaped
grooves. This method involves problems in
controllability and productivity in deposition of
5   polycrystalline Si in a thickness of as large as
several hundred microns, and in removal of the
monocrystalline Si substrate by grinding at the back
face to leave an isolated active Si layer only.

2. An SiO_2 layer is formed by ion implantation into
10   a monocrystalline Si substrate (SIMOX: Separation by
ion implanted oxygen). This is the most highly
advanced method in view of the matching with the Si
process. This method, however, requires implantation
of oxygen ions in an amount of as much as 10^{18} ions/cm^2,
15   which takes a long time, resulting in low productivity
and high wafer cost. Further, the product has many
remaining crystal defects, and does not have
satisfactory properties for industrial production of a
minority carrier device.

20   3. An SOI structure is formed by oxidation of
porous Si for dielectric isolation. In this method, an
N-type Si layer is formed in an island-like pattern on
a P-type monocrystalline Si substrate surface by proton
ion implantation (Imai, et al.: J. Crystal Growth, Vol.
25   63, p. 547 (1983)) or by epitaxial growth and
patterning, and subsequently only the P-type Si
substrate is made porous by anodic oxidation in an HF

solution to surround the island-patterned N-type Si, and the N-type Si island is dielectrically isolated by accelerated oxidation. In this method, the isolated Si regions are fixed prior to the device process, which

5 may limit the freedom of device design disadvantageously.

4. Differently from the above conventional SOI formation, a method has recently come to be noticed in which a monocrystalline Si substrate is bonded to

10 another thermally oxidized monocrystalline Si substrate by heat treatment or use of an adhesive to form an SOI structure. This method requires uniform thinness of the active layer for the device: namely, formation of a film of a micron thick or thinner from a

15 monocrystalline substrate of several hundred micron thick. This thin film may be formed by either of the two methods below.

1. Thin film formation by grinding, and

2. Thin film formation by selective etching.

20 The grinding method of "1" does not give readily a uniform thin film. In particular, formation of a film of submicron thickness results in thickness variation of tens of percent. This irregularity is a serious problem. With a larger diameter of the wafer,

25 the uniformity of the thickness is much more difficult to attain.

The etching method of "2" is regarded to be

effective for uniform thin film formation. This method, however, involves the problems of insufficient selectivity of about 10^2 at the highest, inferior surface properties after etching, and low crystallinity of the SOI layer because of the employed ion implantation, epitaxial or heteroepitaxial growth on a high-concentration B-doped Si layer. (C. Harendt, et al.: J. Elect. Mater., Vol. 20, p. 267 (1991); H. Baumgart, et al.: Extended Abstract of ECS 1st International Symposium of Wafer Bonding, pp. 733- (1991); and C.E. Hunt: Extended Abstract of ECS 1st International Symposium of Wafer Bonding, pp. 696- (1991))

The semiconductor substrate which is prepared by lamination requires two wafers essentially, and the most part of the one of the wafers is discarded by grinding or etching, thereby wasting the resource. Therefore the SOI prepared by lamination involves many problems in controllability, uniformity, production cost, and so forth in conventional processes.

A thin Si layer deposited on a light-transmissive substrate typified by a glass plate becomes amorphous or polycrystalline owing to disorder of crystallinity of the substrate, not giving high performance of the device. Simple deposition of Si does not give desired quality of single crystal layer owing to the amorphous crystal structure of the

substrate.

The light-transmissive substrate is essential for construction of a light-receiving element such as a contact sensor, and projection type of liquid crystal image-displaying apparatus. Additionally, a driving element of high performance is necessary for higher density, higher resolution, and higher precision of the sensor and of the image elements of the display. Consequently, the element provided on a light transmissive substrate is also required to have monocrystalline layer of high crystallinity.

Amorphous Si or polycrystalline Si will not give a driving element having required sufficient performance because of the many defects in the crystal structure.

As mentioned above, a compound semiconductor device requires essentially a compound semiconductor substrate. The compound semiconductor substrate, however, is expensive, and is not readily formed in a larger size.

Epitaxial growth of a compound semiconductor such as GaAs on an Si substrate gives a grown film of poor crystallinity owing to the difference in the lattice constants and the thermal expansion coefficients, thereby the resulting grown film being not suitable for use for a device.

Epitaxial growth of a compound semiconductor on

porous Si is intended for mitigation of misfit of the lattices. However, the substrate has not sufficient stability and reliability owing to the low thermal stability and long-term deterioration of the porous Si.

5   In view of the above-mentioned problems, Takao Yonehara, one of the inventors of the present invention, disclosed formerly a novel process for preparing a semiconductor member in European Patent Publication No. 0469630A2. This process comprises the

10 steps of forming a member having a nonporous monocrystalline semiconductor region on a porous monocrystalline semiconductor region; bonding the surface of a member of which the surface is constituted of an insulating substance onto the surface of the

15 nonporous monocrystalline semiconductor region; and then removing the porous monocrystalline semiconductor region by etching. This process is satisfactory for solving the above-mentioned problems. Further improvement of the disclosed process for higher

20 productivity and lower production cost will contribute greatly to the industries concerned.

SUMMARY OF THE INVENTION

    The present invention intends to improve

25 further the process disclosed in the above European Patent for producing a semiconductor member.

    The present invention further intends to

provide a process for producing economically a semiconductor substrate having a monocrystalline layer or a compound semiconductor monocrystalline layer having excellent crystallinity, large-area and uniform

5   flat surface on a surface of a monocrystalline substrate, in which the substrate is removed to leave the active semiconductor layer to obtain a monocrystalline layer or a compound semiconductor monocrystalline layer formed on the surface and having

10   few defects.

The present invention still further intends to provide a process for producing a semiconductor substrate on a transparent substrate (light-transmissive substrate) for obtaining a monocrystalline

15   Si semiconductor layer or a monocrystalline compound semiconductor layer having crystallinity as high as that of a monocrystalline wafer with high productivity, high uniformity, high controllability, and low production cost.

20   The present invention still further intends to provide a process for producing a semiconductor substrate useful in place of expensive SOS or SIMOX in production of a large scale integrated circuit of SOI structure.

25   A first embodiment of the process for producing a semiconductor substrate of the present invention comprises steps of: forming a nonporous monocrystalline

semiconductor layer on a porous layer of the first substrate having the porous layer; bonding the nonporous monocrystalline layer onto a second substrate; separating the bonded substrates at the

5   porous layer; removing the porous layer on the second substrate; and removing the porous layer constituting the first substrate.

A second embodiment of the process for producing a semiconductor substrate of the present

10   invention comprises steps of: forming a nonporous monocrystalline semiconductor layer on a porous layer of a first substrate having the porous layer; bonding the nonporous monocrystalline layer onto a second substrate with interposition of an insulative layer;

15   separating the bonded substrates at the porous layer; removing the porous layer on the second substrate; and removing the porous layer constituting the first substrate.

In the present invention, the lamination-bonded

20   substrates are separated at the porous layer, and the porous layer is removed from the second substrate having a nonporous monocrystalline semiconductor layer. Thereby, a semiconductor substrate is prepared which has nonporous monocrystalline semiconductor layer of

25   high quality. Furthermore, the first substrate can be repeatedly used for producing the semiconductor substrate in the next production cycle by removing the

remaining porous layer on the first substrate after the separation of the two substrate. Thereby, the semiconductor substrate can be produced with higher productivity and lower cost.

5   The present invention enables preparation of a monocrystalline layer of Si or the like, or a monocrystalline compound semiconductor layer having excellent crystallinity similar to monocrystalline wafers on a substrate including a light-transmissive

10   substrate with advantages in productivity, uniformity, controllability, an production cost.

The present invention further enables production of a semiconductor substrate which can be a substitute for expensive SOS and SIMOX in production of

15   large scale integrated circuits of an SOI structure.

According to the present invention, the combined substrates are separated at the porous layer or layers into two or more substrates, and the one or more separated substrates may be used as a

20   semiconductor substrate after removal of the remaining porous layer, and the other substrate may be used repeatedly in the next production cycle of a semiconductor substrate.

Further, according to the present invention,

25   two semiconductor substrates can be produced simultaneously by forming porous layers and nonporous monocrystalline layers on the both faces of a

substrate, bonding thereto two other substrates, and separating the substrates at the porous layer.

BRIEF DESCRIPTION OF THE DRAWINGS

5   Figs. 1A to 1E are schematic drawings for explaining an example of the process of the present invention.

Figs. 2A to 2E are schematic drawings for explaining another example of the process of the

10   present invention.

Figs. 3A to 3E are schematic drawings for explaining a still another example of the process of the present invention.

Figs. 4A to 4E are schematic drawings for

15   explaining a further example of the process of the present invention.

Figs. 5A to 5E are schematic drawings for explaining a still further example of the process of the present invention.

20

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The process for producing a semiconductor substrate of the present invention is described by employing a silicon substrate as an example.

25   The mechanical strength of porous silicon is much lower than that of bulk silicon depending on the porosity thereof. For instance, porous silicon having

a porosity of 50% is considered to have half a
mechanical strength of bulk silicon. Therefore, on
application of a tensile force, a compressive force, or
a shearing force to a laminated wafer, the porous layer

5   will be firstly broken. The larger the porosity of the
porous layer, the less is the force for the breakdown
of the layer.

A silicon substrate can be made porous by
anodization in an HF solution. The resulting porous Si

10   layer has a density ranging from 1.1 to 0.6 g/cm^3
depending on the HF solution concentration of from 50
to 20% in comparison with the density of 2.33 g/cm^3 of
monocrystalline Si. The porous layer is formed only on
a P-type Si substrate, but is not formed on an N-type

15   Si layer by the reasons described later. The porous Si
layer has pores of about 600 Å in average diameter
according to transmissive electron microscopy.

The porous Si was found by Uhlir, et al. in the
year 1956 during the study of electropolishing of

20   semiconductors (A. Uhlir: Bell Syst. Tech. J., vol. 35,
p. 333 (1956)).

Unagami, et al. found that positive holes are
required for anodization of Si in an HF solution, and
the reactions proceed as shown in their report on

25   dissolution of Si in anodization (T. Unagami, et al.:
J. Electrochem. Soc., vol. 127, p. 476 (1980)) as
below:

$$Si + 2HF + (2-n)e^+ \rightarrow SiF_2 + 2H^+ + ne^-$$

$$SiF_2 + 2HF \rightarrow SiF_4 + H_2$$

$$SiF_4 + 2HF \rightarrow H_2SiF_6$$

or

5

$$Si + 4HF + (2-\lambda)e^+ \rightarrow SiF_4 + 4H^+ + \lambda e^-$$

$$SiF_4 + 2HF \rightarrow H_2SiF_6$$

where e^+ and e^- represent respectively a positive hole and an electron; n and λ represent respectively the number of positive holes required for dissolving one Si

10 atom. Unagami reported that porous Si is formed under the condition of $n > 2$, or $\lambda > 4$.

According to the above consideration, P-type Si which has positive holes can be made porous, whereas N-type Si cannot be made porous. This selectivity for

15 porosity was evidenced by Nagano, et al., and Imai (Nagano, Nakajima, Yasuno, Oonaka, and Kajihara: Denshi Tsushin Gakkai Gijutsu Kenkyu Hokoku (Technical Research Report of Electronic Communication Society) vol. 79, SSD79-9549 (1979); and K. Imai: Solid-State

20 Electronics, vol. 24, p. 159 (1981)).

On the other hand, a report is found that high concentration N-type Si can be made porous (R.P. Holmstrom and J.Y. Chi: Appl. Phys. Lett., vol. 42, p. 386 (1983)). Therefore, selection of the substrate is

25 important for producing porous Si regardless of P-type or N-type.

The porous Si layer has pores of about 600 Å in

average diameter by observation by transmission

electron microscopy, and the density is less than half

that of monocrystalline Si. Nevertheless, the single

crystallinity is maintained, and thereon a

5   monocrystalline Si can be made to grow epitaxially in a

layer. However, in the epitaxial growth at a

temperature of 1000°C or higher, the internal pores

will come to be rearranged, which impairs the

accelerated etching characteristics. Therefore, low

10   temperature growth processes are preferred for

epitaxial growth of the Si layer, such as molecular

beam epitaxial growth, plasma CVD, reduced pressure

CVD, photo-assisted CVD, bias sputtering, and liquid-

phase epitaxial growth.

15   The porous layer has a large volume of voids

therein, having a half or lower density of the

material, and having a surface area remarkably large

for the volume. Accordingly, the chemical etching is

greatly accelerated in comparison with that of the

20   normal monocrystalline layer.

Embodiment 1

A first monocrystalline Si substrate 11 is made

porous at the surface to form a porous layer 12 as

shown in Fig. 1A. Then, nonporous monocrystalline Si

25   layer 13 is formed on the porous Si layer 12 as shown

in Fig. 1B.

Another Si supporting substrate 14 is brought

into contact with the nonporous monocrystalline Si layer 13 with interposition of an insulative layer 15 at room temperature as shown in Fig. 1C, and then the contacted matter was subjected to anode coupling,

5   compression, heat treatment, or combination thereof to bond tightly the Si supporting substrate 14 and the monocrystalline layer 13 with interposition of the insulative layer 15. The insulative layer 15 may be formed preliminarily on either one of the

10   monocrystalline Si layer 13 or the Si supporting substrate 14, or the three sheets may be bonded with an insulative thin film interposed.

Subsequently, the substrates are separated at the porous Si layer 12 as shown in Fig. 1D. On the Si

15   supporting substrate 14, the layers have the structure of porous Si 12 / monocrystalline Si layer 13 / insulative layer 15 / Si supporting substrate 14.

The porous Si 12 is removed selectively by non-electrolytic wet chemical etching by use of at least

20   one of a usual Si etching solution, hydrofluoric acid or a mixture of hydrofluoric acid with alcohol and/or hydrogen peroxide as the porous Si-selective etching solution, and buffered hydrofluoric acid or a mixture of hydrofluoric acid with alcohol and/or hydrogen

25   peroxide to leave the thin-layered monocrystalline Si layer 13 on the insulative substrate 15+14. As described above in detail, the porous Si can be etched

selectively by a usual Si etching solution owing to the extremely large surface area of the porous surface area.

Otherwise, the porous Si 12 is selectively removed by grinding by utilizing the monocrystalline Si layer 13 as the grinding stopper.

Fig. 1E illustrates a semiconductor substrate of the present invention. The monocrystalline Si layer 13 is formed flat and uniformly in a thin layer on the insulative substrate 15+14 over the entire large area of the wafer. The obtained semiconductor substrate is useful for production of insulation-isolated electronic elements.

The first monocrystalline Si substrate 11 may be repeatedly used for the same use after removal of the remaining Si and surface flattening treatment if the surface has become roughened unacceptably in the next production cycle.

The method of separation of the two substrates at the porous Si layer in the present invention includes crushing of the porous layer by compression on the both faces of the bonded substrates; pulling of the respective substrates in opposite directions; insertion of a jig or the like into the porous layer; application of force in opposite directions parallel to the bonded face of the substrates; application of supersonic vibration to the porous layer; and so forth.

The porosity of the porous Si layer suitable for the separation ranges generally from 10 to 80%, preferably from 20 to 60%.

Embodiment 2

5   A first monocrystalline Si substrate 21 is made porous at the surface to form a porous layer 22 as shown in Fig. 2A. Then a nonporous monocrystalline Si layer 23 is formed on the porous Si layer 22 as shown in Fig. 2B.

10   A light-transmissive supporting substrate 24 is brought into contact with the monocrystalline Si layer 23 with interposition of an insulative layer 25 at room temperature as shown in Fig. 2C, and then the contacted matter was subjected to anode coupling, compression,

15   heat treatment, or combination of the treatment to bond tightly the light-transmissive supporting substrate 24 and the monocrystalline layer 23 with interposition of the insulative layer 25. The insulative layer 25 may be formed preliminarily on either one of the

20   monocrystalline Si layer or the light-transmissive supporting substrate 24, or the three sheets may be bonded with interposition of an insulative thin film.

       Subsequently, the substrates are separated at the porous Si layer 22 as shown in Fig. 2D. On the

25   light-transmissive supporting substrate, the layers have the structure of porous Si 22 / monocrystalline Si layer 23 / insulative layer 25 / light-transmissive

supporting substrate 24.

The porous Si 22 is removed selectively by non-electrolytic wet chemical etching by use of at least one of a usual Si etching solution, hydrofluoric acid

5   or a mixture of hydrofluoric acid with alcohol and/or hydrogen peroxide as the porous Si-selective etching solution, and buffered hydrofluoric acid or a mixture of hydrofluoric acid with alcohol and/or hydrogen peroxide to leave a thin-layered monocrystalline Si

10   layer 23 on the insulative substrate 25+24. As described above in detail, the porous Si can be etched selectively by a usual Si etching solution because of the extremely large surface area of the porous surface area.

15   Otherwise, the porous Si 23 is selectively removed by grinding by utilizing the monocrystalline Si layer 22 as the grinding stopper.

Fig. 2E illustrates a semiconductor substrate of the present invention. The monocrystalline Si layer

20   23 is formed flat and uniformly in a thin layer on the insulative substrate 25+24 over the entire large area of the wafer. The obtained semiconductor substrate is useful for production of insulation-isolated electronic elements.

25   The presence of the interposed insulative layer 25 is not essential.

The first monocrystalline Si substrate 21 may

be repeatedly used for the same use after removal of
the remaining Si and surface flattening treatment if
the surface has become roughened unacceptably in the
next production cycle.

5   Embodiment 3

A first monocrystalline Si substrate 31 is made
porous at the surface to form a porous layer 32 as
shown in Fig. 3A. Then a nonporous monocrystalline
compound semiconductor layer 33 is formed on the porous
10   Si layer 32 as shown in Fig. 3B.

Another Si supporting substrate 34 is brought
into close contact with the monocrystalline compound
semiconductor layer 33 with interposition of an
insulative layer 35 at room temperature as shown in
15   Fig. 3C, and then the contacted matter was subjected to
anode coupling, compression, or heat treatment, or
combination of the treatments to bond tightly the Si
supporting substrate 34 and the monocrystalline layer
33 with interposition of the insulative layer 35. The
20   insulative layer 35 may be formed preliminarily on
either one of the monocrystalline compound
semiconductor layer or the Si supporting substrate 34,
or the three sheets may be bonded with interposition of
an insulative thin film.

25   Subsequently, the substrates are separated at
the porous Si layer 32 as shown in Fig. 3D. On the Si
supporting substrate, the layers have the structure of

porous Si 32 / monocrystalline compound semiconductor
layer 33 / insulative layer 35 / Si supporting
substrate 34.

The porous Si 32 is removed selectively by

5   chemical etching by use of an etching solution which is
capable of etching Si at a higher etching rate than the
compound semiconductor to leave the thin-layered
monocrystalline compound semiconductor layer 33 on the
insulative substrate 35+34.

10   Otherwise, the porous Si 32 is selectively
removed by grinding by utilizing the monocrystalline
compound semiconductor layer 32 as the grinding
stopper.

Fig. 3E illustrates a semiconductor substrate

15   of the present invention. The monocrystalline compound
semiconductor layer 33 is formed flat and uniformly in
a thin layer on the insulative substrate 35+34 over the
entire large area of the wafer. The obtained
semiconductor substrate is useful as a compound

20   semiconductor substrate and for production of
insulation-isolated electronic elements.

When the substrate is used as a compound
semiconductor substrate, the insulative layer 35 is not
essential.

25   The first monocrystalline Si substrate 31 may
be repeatedly used for the same use after removal of
the remaining Si and surface flattening treatment if

the surface has become roughened unacceptably in the next production cycle.

Embodiment 4

A first monocrystalline Si substrate 41 is made porous at the surface to form a porous layer 42 as shown in Fig. 4A. Then a nonporous monocrystalline compound semiconductor layer 43 is formed on the porous Si layer 42 as shown in Fig. 4B.

A light-transmissive supporting substrate 44 is brought into close contact with the monocrystalline compound semiconductor layer 43 with interposition of an insulative layer 45 at room temperature as shown in Fig. 4C, and then the contacted matter was subjected to anode coupling, compression, heat treatment, or combination of the treatments to bond tightly the light-transmissive supporting substrate 44 with the monocrystalline layer 43 with interposition of the insulative layer 45. The insulative layer 45 may be formed preliminarily on either one of the monocrystalline compound semiconductor layer or the light-transmissive supporting substrate 44, or the three sheets may be bonded with interposition of an insulative thin film.

Subsequently, the substrates are separated at the porous Si layer 42 as shown in Fig. 4D. On the light-transmissive supporting substrate, the layers have the structure of porous Si 42 / monocrystalline

compound semiconductor layer 43 / insulative layer 45 / light-transmissive supporting substrate 44.

The porous Si 42 is removed selectively by chemical etching by use of an etching solution which is capable of etching Si at a higher etching rate than the compound semiconductor to leave a thin-layered monocrystalline compound semiconductor layer 43 on the insulative substrate 45+44.

Otherwise, the porous Si 42 is selectively removed by grinding by utilizing the monocrystalline compound semiconductor layer 42 as the grinding stopper.

Fig. 4E illustrates a semiconductor substrate of the present invention. The monocrystalline compound semiconductor layer 43 is formed flat and uniformly in a thin layer on the insulative substrate 45+44 over the entire large area of the wafer. The obtained semiconductor substrate is useful for production of insulation-isolated electronic elements.

The insulative layer 45 is not essential in this embodiment.

The first monocrystalline Si substrate 41 may be repeatedly used for the same use after removal of the remaining Si and surface flattening treatment if the surface has become roughened unacceptably in the next production cycle.

Embodiment 5

A first monocrystalline Si substrate 51 is made
porous at the both faces to form porous layers 52, 53
as shown in Fig. 5A. Then, nonporous monocrystalline
compound semiconductor layers 54, 55 are formed on the
5   porous Si layers 52, 53 as shown in Fig. 5B.

Two supporting substrates 56, 57 are brought
into close contact with the monocrystalline
semiconductor layers 54, 55 with interposition of
insulative layers 58, 59 respectively at room
10   temperature as shown in Fig. 5C, and then the contacted
matter is subjected to anode coupling, compression,
heat treatment, or combination of the treatments to
bond tightly the supporting substrates 56, 57 and the
monocrystalline layers 54, 55 with interposition of the
15   insulative layers 58, 59. In the bonding, the
respective insulative layers 58, 59 may be formed
preliminarily on either one of the monocrystalline
semiconductor layer 54, 55 or the supporting substrate
56, or the five sheets may be bonded with interposition
20   of insulative thin films.

Subsequently, the substrates are separated into
three at the both porous Si layers 52, 53 as shown in
Fig. 5D. The two supporting substrates come to have a
structure of porous Si / monocrystalline semiconductor
25   layer / insulative layer / supporting substrate
(52/54/58/56, and 53/55/59/57).

The porous Si layers 52, 53 are removed

selectively by chemical etching to leave thin-layered monocrystalline semiconductor layers 54, 55 on the supporting substrates 58/56 and 59/57.

Otherwise, the porous Si 52, 53 is selectively

5 removed by grinding by utilizing the monocrystalline semiconductor layers 54, 55 as the grinding stopper.

Fig. 5E illustrates semiconductor substrates prepared according to the present invention. The monocrystalline compound semiconductor layers are

10 formed flat and uniformly in a thin layer on the supporting substrates over the entire large area of the two wafers at a time with a large area. The obtained semiconductor substrate is useful for production of insulation-isolated electronic elements.

15 The insulative intervening layers 58, 59 are not essential.

The supporting substrates 56, 57 need not be the same.

The first monocrystalline Si substrate 51 may

20 be repeatedly used for the same use after removal of the remaining Si and surface flattening treatment if the surface has become roughened unacceptably in the next production cycle.

Example 1

25 A first monocrystalline (100) Si substrate of P-type having a diameter of 6 inches, a thickness of 625 μm, and a specific resistance of 0.01 Ω·cm was

anodized in an HF solution under the anodization
conditions as below:

Current density: 5 mA·cm^{-2}

Anodization solution: $HF:H_2O:C_2H_5OH = 1:1:1$

5   Time: 12 minutes

Thickness of porous Si: 10 μm

Porosity: 15 %

This substrate was oxidized at 400°C in an
oxygen atmosphere for one hour. Thereby the inner wall
10   of the pores of the porous Si was covered with a
thermal oxidation film. On the porous Si,
monocrystalline Si was allowed to grow epitaxially in a
thickness of 1 μm by CVD (chemical vapor deposition)
under the growth conditions below:

15   Source gas: SiH_2Cl_2/H_2

Gas flow rate: 0.5/180 ℓ/min

Gas pressure: 80 Torr

Temperature: 950°C

Growth rate: 0.3 μm/min

20   The face of the epitaxially grown Si layer was
thermally oxidized to form an SiO_2 layer of 100 nm
thick.

On the face of this Si substrate, a separately
prepared second Si substrate having an SiO_2 layer of 500
25   nm thick was superposed with the SiO_2 layer inside, and
the superposed matter was heat-treated at 900°C for 2
hours to bond the substrates tightly.

A pulling force was applied to the resulting bonded wafer in the direction perpendicular to the wafer face in such a manner that a plate was bonded respectively to each of the both faces of the wafer

5 with an adhesive and the plates were pulled to opposite directions with a jig. Consequently, the porous Si layer was broken to cause separation of the wafer into two sheets with the porous Si layers exposed.

The porous Si layer on the second substrate was

10 etched selectively in a mixture of 49% hydrofluoric acid and 30% hydrogen peroxide (1:5) with agitation. The porous Si was etched and removed completely with the monocrystalline Si remaining unetched as an etching stopper. The etching rate of the nonporous

15 monocrystalline Si was extremely low, the selection ratio of the etching rate of the porous Si being 10^5 or higher. Therefore, thickness decrease of the nonporous layer by etching was practicably negligible (several ten $\overset{\circ}{A}$).

20 Consequently, a monocrystalline Si layer was formed in a thickness of 1 µm on an Si oxide film. The monocrystalline Si layer did not change at all by the selective etching of the porous Si layer.

The cross-section of the Si layer was observed

25 by transmission electron microscopy, and it was confirmed that no additional crystal defect was formed in the Si layer and the excellent crystallinity was

retained.

Thus an SOI substrate was obtained which has a
semiconductor layer of high quality.

The other Si substrate having been separated at
5   the porous Si layer portion was etched in the same
manner as above to remove the remaining porous layer,
and its surface was polished. The obtained Si
substrate was used repeatedly for the same use in the
next production cycle. Thereby a plurality of SOI
10   substrates having a semiconductor layer of high quality
were obtained.

Example 2

A first monocrystalline (100) Si substrate of
P-type having a diameter of 4 inches, a thickness of
15   525 μm, and a specific resistance of 0.01 Ω•cm was
anodized in an HF solution under the anodization
conditions as below:

Current density: 7 mA•cm^{-2}

Anodization solution: HF:H$_2$O:C$_2$H$_5$OH = 1:1:1

20   Time: 12 minutes

Thickness of porous Si: 10 μm

Porosity: 15 %

This substrate was oxidized at 400°C in an
oxygen atmosphere for 2 hours. Thereby the inner wall
25   of the pores of the porous Si was covered with a
thermal oxidation film. On the porous Si,
monocrystalline Si was allowed to grow epitaxially in a

thickness of 0.5 μm by MBE (molecular beam epitaxy) under the growth conditions below:

Temperature:	700°C
Pressure:	1×10^{-9} Torr
Growth rate:	0.1 nm/sec
Temperature:	950°C
Growth rate:	0.3 μm/min

5

The face of the epitaxially grown Si layer was thermally oxidized to form an SiO_2 layer of 100 nm

10   thick.

On the face of the SiO_2 layer, was superposed a separately prepared fused quartz substrate, and the superposed matter was heat-treated at 400°C for 2 hours to bond the substrates.

15   A sufficient compression force was applied uniformly to the resulting bonded wafer in the direction perpendicular to the wafer face such that plates were bonded to each of the both faces of the wafer with an adhesive and the compression force was

20   applied with the same jig as in Example 1.

Consequently, the porous Si layer was broken to cause separation of the wafer into two sheets with the porous Si layers exposed.

The porous Si layers were etched selectively in

25   a mixture of buffered hydrofluoric acid and 30% hydrogen peroxide (1:5) with agitation. Thereby the porous Si was etched and removed completely with the

monocrystalline Si remaining unetched as an etch-stop material. The etching rate of the nonporous monocrystalline Si was extremely low, the selection ratio of the etching rate of the porous Si being, 10^5 or higher. Therefore, thickness decrease of the nonporous layer by etching was practicably negligible (several ten $\overset{\circ}{A}$).

Consequently, a monocrystalline Si layer was formed in a thickness of 0.5 μm on a fused quartz substrate. The monocrystalline Si layer did not change at all by the selective etching of the porous Si layer.

The cross-section of the Si layer was observed by transmission electron microscopy, and it was confirmed that no additional crystal defect was formed in the Si layer and the excellent crystallinity was retained.

A plurality of SOI substrates having a semiconductor layer of high quality were prepared by repeating the above process in the same manner as in Example 1.

Example 3

A first monocrystalline (100) Si substrate of P-type or N-type having a diameter of 6 inches, a thickness of 625 μm, and a specific resistance of 0.01 Ω•cm was anodized in an HF solution under the anodization conditions as below:

Current density: 7 mA•cm^{-2}

Anodization solution: HF:H_2O:C_2H_5OH = 1:1:1

Time: 12 minutes

Thickness of porous Si: 10 μm

Porosity: 15 %

5   This substrate was oxidized at 400°C in an

oxygen atmosphere for one hour. Thereby the inner wall

of the pores of the porous Si was covered with a

thermal oxidation film. On the porous Si,

monocrystalline GaAs was allowed to grow epitaxially in

10   a thickness of 1 μm by MOCVD (metal organic chemical

vapor deposition) under the growth conditions below:

Source gas: TMG / AsH_3 / H_2

Gas pressure: 80 Torr

Temperature: 700°C

15   On the face of the formed GaAs layer, was

superposed a separately prepared second Si substrate,

and the superposed matter was heat-treated at 900°C for

one hour to bond the substrates tightly.

A sufficient compression force was applied to

20   the resulting bonded wafer in the same manner as in

Example 2. Thereby, the porous Si layer was broken to

allow the wafer to separate into two sheets with the

porous Si layers exposed.

Then, the oxide film on the inner wall of the

25   porous Si layer was removed by hydrofluoric acid, and

the porous Si was etched with a mixture of ethylene

diamine, pyrocathecol, and water (17 ml : 3 g : 8 ml)

at 110°C. Thereby the porous Si was etched selectively
and removed off completely with the monocrystalline
GaAs remaining unetched as an etch-stopping material.
The etching rate of the nonporous monocrystalline GaAs
5   was extremely low and practicably negligible.

Consequently, a monocrystalline GaAs layer was
formed in a thickness of 1 μm on an Si substrate. The
monocrystalline GaAs layer did not change at all by the
selective etching of the porous Si layer.

10   The cross-section of the GaAs layer was
observed by transmission electron microscopy, and it
was confirmed that no additional crystal defect was
formed in the GaAs layer and the excellent
crystallinity was retained.

15   A plurality of semiconductor substrates having
a GaAs layer of high quality were prepared by repeating
the above process in the same manner as in Example 2.

GaAs on an insulative film was also prepared by
employing an Si substrate having an oxide film as the
20   supporting substrate.

Example 4

A first monocrystalline (100) Si substrate of
P-type or N-type having a diameter of 5 inches, a
thickness of 625 μm, and a specific resistance of 0.01
25   Ω•cm was anodized in an HF solution under the
anodization conditions as below:

Current density: 10 mA•cm^{-2}

Anodization solution: $HF:H_2O:C_2H_5OH = 1:1:1$

Time: 24 minutes

Thickness of porous Si: 20 µm

Porosity: 17 %

5   This substrate was oxidized at 400°C in an

oxygen atmosphere for 2 hours. Thereby the inner wall

of the pores of the porous Si was covered with a

thermal oxidation film. On the porous Si,

monocrystalline AlGaAs was allowed to grow epitaxially

10   in a thickness of 0.5 µm by MBE (molecular beam

epitaxy).

On the face of the formed AlGaAs layer, was

superposed a face of a separately prepared low-melting

glass substrate. The superposed matter was heat-

15   treated at 500°C for 2 hours to bond the substrates

tightly.

A sufficient compression force was applied to

the resulting bonded wafer in the same manner as in

Example 2. Thereby, the porous Si layer was broken to

20   allow the wafer to separate into two sheets with the

porous Si layers exposed.

The porous Si was etched with hydrofluoric acid

solution. Thereby the porous Si was etched selectively

and removed off completely with the monocrystalline

25   AlGaAs remaining unetched as an etch-stopping material.

The etching rate of the nonporous monocrystalline

AlGaAs was extremely low and practicably negligible.

Consequently, a monocrystalline AlGaAs layer was formed in a thickness of 0.5 μm on a glass substrate. The monocrystalline AlGaAs layer did not change at all by the selective etching of the porous Si

5   layer.

The cross-section of the AlGaAs layer was observed by transmission electron microscopy, and it was confirmed that no additional crystal defect was formed in the AlGaAs layer and the excellent

10   crystallinity was retained.

A plurality of semiconductor substrates having a GaAs layer of high quality were prepared by repeating the above process in the same manner as in Example 2.

Example 5

15   A first monocrystalline (100) Si substrate of P-type or N-type having been polished on the both faces and having a diameter of 6 inches, a thickness of 625 μm, and a specific resistance of 0.01 Ω•cm was anodized on the both faces in an HF solution under the

20   anodization conditions below:

Current density: 5 mA•cm^{-2}

Anodization solution: HF:H$_2$O:C$_2$H$_5$OH = 1:1:1

Time: 12 × 2 minutes

Thickness of porous Si: 10 μm each

25   Porosity: 15 %

This substrate was oxidized at 400°C in an oxygen atmosphere for one hour. Thereby the inner wall

of the pores of the porous Si was covered with a

thermal oxidation film. On the porous Si formed on the

both faces of the substrate, monocrystalline Si was

allowed to grow epitaxially in a thickness of 1 μm

5   respectively by CVD (chemical vapor deposition) under

the growth conditions below:

Source gas: SiH_2Cl_2/H_2

Gas flow rate: 0.5/180 ℓ/min

Gas pressure: 80 Torr

10   Temperature: 950°C

Growth rate: 0.3 μm/min

The faces of the formed epitaxial Si layers

were thermally oxidized to form SiO_2 layers in a

thickness of 100 nm.

15   On each of the faces of the SiO_2 layers, a

separately prepared Si substrate having a 500-nm thick

SiO_2 layer was superposed respectively with the SiO_2

layers inside, and the superposed matter was heat-

treated at 600°C for 2 hours to bond the substrates

20   tightly.

A sufficient pulling force was applied to the

resulting bonded wafer in the direction perpendicular

to the bonded wafer face in the same manner as in

Example 1. Thereby, the two porous Si layers were

25   broken to allow the wafer to separate into three sheets

with the porous Si layers exposed.

The porous Si layers were etched selectively

with a mixture of 49% hydrofluoric acid with 30%

hydrogen peroxide (1:5) with agitation. Thereby the

porous Si was etched selectively and removed completely

with the monocrystalline Si remaining unetched as an

5   etch-stopping material. The etching rate of the

nonporous monocrystalline Si was extremely low, the

selection ratio of the etching rate of the porous Si

being 10^5 or higher. Therefore, thickness decrease of

the nonporous layer by etching was practicably

10   negligible (several ten Å).

Consequently, a monocrystalline Si layer was

formed in a thickness of 1 μm respectively on the two

Si oxide films simultaneously. The monocrystalline Si

layers did not change at all by the selective etching

15   of the porous Si layer.

The cross-section of the Si layer was observed

by transmission electron microscopy, and it was

confirmed that no additional crystal defect was formed

in the Si layer and the excellent crystallinity was

20   retained.

A plurality of semiconductor substrates having

a semiconductor layer of high quality were prepared by

repeating the above process in the same manner as in

Example 1.

25   Example 6

A first monocrystalline (100) Si substrate of

P-type or N-type having a diameter of 5 inches, a

thickness of 625 μm, and a specific resistance of 0.01
Ω·cm was anodized in an HF solution under the
anodization conditions below:

<div style="margin-left:2em">

Current density:	7 mA·cm^{-2}
Anodization solution:	HF:H$_2$O:C$_2$H$_5$OH = 1:1:1
Time:	4 minutes
Thickness of porous Si:	3 μm
Porosity:	15 %

</div>

The anodization was conducted further under the
conditions below:

<div style="margin-left:2em">

Current density:	30 mA·cm^{-2}
Anodization solution:	HF:H$_2$O:C$_2$H$_5$OH = 1:3:2
Time:	3 minutes
Thickness of porous Si:	10 μm
Porosity:	45 %

</div>

This substrate was oxidized at 400°C in an
oxygen atmosphere for one hour. Thereby the inner wall
of the pores of the porous Si was covered with a
thermal oxidation film. On the porous Si formed on the
substrate, monocrystalline Si was allowed to grow
epitaxially in a thickness of 0.3 μm by CVD under the
conditions below:

<div style="margin-left:2em">

Source gas:	SiH$_4$
Carrier gas:	H$_2$
Temperature:	850°C
Pressure:	1 × 10^{-2} Torr
Growth rate:	3.3 nm/sec

</div>

The surface of the formed epitaxial Si layer was thermally oxidized to form SiO_2 layer in a thickness of 100 nm.

On the face of the SiO_2 layer, a separately prepared second Si substrate having a 500-nm thick SiO_2 layer was superposed with the SiO_2 layer inside, and the superposed matter was heat-treated at 700°C for 2 hours to bond the substrates tightly.

A sufficient pulling force was applied to the resulting bonded wafer in the direction perpendicular to the bonded wafer face in the same manner as in Example 1. Thereby, the porous Si layer was broken to allow the wafer to separate into two sheets with the porous Si layers exposed.

The porous Si on the second Si substrate was etched selectively with an etching solution of $HF/HNO_3/CH_3COOH$ type. Thereby the porous Si was etched selectively and removed completely. The etching rate of the nonporous monocrystalline Si was extremely low, so that the thickness decrease of the nonporous layer by etching was practicably negligible.

Consequently, a monocrystalline Si layer was formed in a thickness of 1 μm on the Si oxide film. The monocrystalline Si layers did not change at all by the selective etching of the porous Si layer.

The cross-section of the Si layer was observed by transmission electron microscopy, and it was

confirmed that no additional crystal defect was formed in the Si layer and the excellent crystallinity was retained.

A plurality of semiconductor substrates having a semiconductor layer of high quality were prepared by repeating the above process in the same manner as in Example 1.

Example 7

A first monocrystalline (100) Si substrate of P-type or N-type having a diameter of 6 inches, a thickness of 625 µm, and a specific resistance of 0.01 Ω·cm was anodized in an HF solution under the anodization conditions below:

Current density: 5 mA·cm^{-2}

Anodization solution: HF:H_2O:C_2H_5OH = 1:1:1

Time: 12 minutes

Thickness of porous Si: 10 µm

Porosity: 15 %

This substrate was oxidized at 400°C in an oxygen atmosphere for one hour. Thereby the inner wall of the pores of the porous Si was covered with a thermal oxidation film. On the porous Si formed on the substrate, monocrystalline Si was allowed to grow epitaxially in a thickness of 1 µm by CVD under the growth conditions below:

Source gas: SiH_2Cl_2/H_2

Gas flow rate: 0.5/180 ℓ/min

Gas pressure: 80 Torr

Temperature: 950°C

Growth rate: 0.3 μm/min

The surface of the formed epitaxial Si layer

5   was thermally oxidized to form SiO_2 layer in a thickness

of 100 nm.

On the face of the SiO_2 layer, a separately

prepared second Si substrate having a 500-nm thick SiO_2

layer was superposed with the SiO_2 layer inside, and the

10   superposed matter was heat-treated at 900°C for 2 hours

to bond the substrates tightly.

A sufficient pulling force was applied to the

resulting bonded wafer in the direction perpendicular

to the bonded wafer face in the same manner as in

15   Example 1.   Thereby, the porous Si layer was broken to

allow the wafer to separate into two sheets with the

porous Si layers exposed.

The porous Si layer on the second substrate was

ground selectively by utilizing the monocrystalline

20   layer as the stopper.   Thereby the porous Si was

removed selectively.

Consequently, a monocrystalline Si layer was

formed in a thickness of 1 μm on the Si oxide film.

The monocrystalline Si layers did not change at all by

25   the selective grinding of the porous Si layer.

The cross-section of the Si layer was observed

by transmission electron microscopy, and it was

confirmed that no additional crystal defect was formed in the Si layer and the excellent crystallinity was retained.

A plurality of semiconductor substrates having a semiconductor layer of high quality were prepared by repeating the above process in the same manner as in Example 1.

Example 8

A first monocrystalline (100) Si substrate of P-type or N-type having a diameter of 6 inches, a thickness of 625 µm, and a specific resistance of 0.01 Ω•cm was anodized in an HF solution under the anodization conditions below:

Current density: $5 \text{ mA} \cdot \text{cm}^{-2}$

Anodization solution: $HF:H_2O:C_2H_5OH = 1:1:1$

Time: 12 minutes

Thickness of porous Si: 10 µm

Porosity: 15 %

This substrate was oxidized at 400°C in an oxygen atmosphere for one hour. Thereby the inner wall of the pores of the porous Si was covered with a thermal oxidation film. On the porous Si formed on the substrate, monocrystalline Si was allowed to grow epitaxially in a thickness of 1 µm by CVD under the conditions below:

Source gas: SiH_2Cl_2/H_2

Gas flow rate: 0.5/180 ℓ/min

Gas pressure: 80 Torr

Temperature: 950°C

Growth rate: 0.3 μm/min

The surface of the formed epitaxial Si layer

5   was thermally oxidized to form SiO_2 layer in a thickness

of 100 nm.

On the face of the SiO_2 layer, a separately

prepared second Si substrate having a 500-nm thick SiO_2

layer was superposed with the SiO_2 layer inside, and the

10   superposed matter was heat-treated at 900°C for 2 hours

to bond the substrates tightly.

A supersonic energy was applied to the

resulting bonded wafer in a vessel provided with a

supersonic oscillator. Thereby, the porous Si layer

15   was broken to allow the wafer to separate into two

sheets with the porous Si layers exposed.

The porous Si layer on the second Si substrate

was etched selectively with a mixture of 49%

hydrofluoric acid with 30% hydrogen peroxide (1:5) with

20   agitation. Thereby the porous Si was etched

selectively and removed completely with the

monocrystalline Si remaining unetched as an etch-

stopping material. The etching rate of the nonporous

monocrystalline Si was extremely low, the selection

25   ratio of the etching rate of the porous Si being 10^5 or

higher. Therefore, thickness decrease of the nonporous

layer by etching was practicably negligible (several

ten Å).

Consequently, a monocrystalline Si layer was formed in a thickness of 1 μm on the Si oxide film. The monocrystalline Si layers did not change at all by the selective etching of the porous Si layer.

The cross-section of the Si layer was observed by transmission electron microscopy, and it was confirmed that no additional crystal defect was formed in the Si layer and the excellent crystallinity was retained.

The first monocrystalline Si substrate was used repeatedly for the same use after removal of the porous Si remaining thereon.

Example 9

A first monocrystalline (100) Si substrate of P-type or N-type having a diameter of 4 inches, a thickness of 525 μm, and a specific resistance of 0.01 $\Omega \cdot cm$ was anodized in an HF solution under the anodization conditions as below:

Current density: 7 $mA \cdot cm^{-2}$

Anodization solution: $HF:H_2O:C_2H_5OH = 1:1:1$

Time: 12 minutes

Thickness of porous Si: 10 μm

Porosity: 15 %

This substrate was oxidized at 400°C in an oxygen atmosphere for 2 hours. Thereby the inner wall of the pores of the porous Si was covered with a

thermal oxidation film. On the porous Si,
monocrystalline Si was allowed to grow epitaxially in a
thickness of 0.5 μm by MBE (molecular beam epitaxy)
under the growth conditions below:

5   Temperature: 700°C

         Pressure: 1×10^{-9} Torr

         Growth rate: 0.1 nm/sec

         Temperature: 950°C

         Growth rate: 0.3 μm/min

10   The surface of the epitaxially grown Si layer
was thermally oxidized to form an SiO_2 layer of 100 nm
thick.

       On the face of the SiO_2 layer, was superposed a
separately prepared fused quartz substrate, and the
15 superposed matter was heat-treated at 400°C for 2 hours
to bond the substrates.

       The end of the porous layer was bared to the
edge face of the wafer, and the porous Si is slightly
etched. Thereto, a sharp blade like a shaver blade
20 was inserted. Thereby, the porous layer was broken,
and the wafer was separated into two sheets with the
porous Si layers exposed.

       The porous Si layer on the fused quartz
substrate was etched selectively in a mixture of
25 buffered hydrofluoric acid and 30% hydrogen peroxide
(1:5) with agitation. Thereby the porous Si was etched
and removed completely with the monocrystalline Si

remaining unetched as an etch-stopping material.   The
etching rate of the nonporous monocrystalline Si was
extremely low, the selection ratio of the etching rate
of the porous Si being, 10^5 or higher.   Therefore,
5   thickness decrease of the nonporous layer by etching
was practicably negligible (several ten Å).

Consequently, a monocrystalline Si layer was
formed in a thickness of 0.5 μm on a fused quartz
substrate.   The monocrystalline Si layer did not change
10   at all by the selective etching of the porous Si layer.

The cross-section of the Si layer was observed
by transmission electron microscopy, and it was
confirmed that no additional crystal defect was formed
in the Si layer and the excellent crystallinity was
15   retained.

The same results were obtained without forming
the oxide film of the surface of the epitaxial Si
surface.

The first monocrystalline Si substrate was used
20   repeatedly for the same use after removal of the
remaining porous Si and mirror-polishing of the
surface.

Example 10

A first monocrystalline (100) Si substrate of
25   P-type or N-type having a polished face on each side
and having a diameter of 6 inches, a thickness of 625
μm, and a specific resistance of 0.01 Ω•cm was anodized

on both sides in an HF solution under the anodization
conditions below:

Current density: 5 mA·cm^{-2}

Anodization solution: HF:H$_2$O:C$_2$H$_5$OH = 1:1:1

Time: 12 × 2 minutes

Thickness of porous Si: 10 µm each

Porosity: 15 %

This substrate was oxidized at 400°C in an
oxygen atmosphere for one hour. Thereby the inner wall
of the pores of the porous Si was covered with a
thermal oxidation film. On the porous Si formed on the
both faces of the substrate, monocrystalline Si was
allowed to grow epitaxially in a thickness of 1 µm by
CVD (chemical vapor deposition) under the conditions
below:

Source gas: SiH$_2$Cl$_2$/H$_2$

Gas flow rate: 0.5/180 ℓ/min

Gas pressure: 80 Torr

Temperature: 950°C

Growth rate: 0.3 µm/min

The surfaces of the formed epitaxial Si layers
were thermally oxidized to form SiO$_2$ layers in a
thickness of 100 nm.

On each of the faces of the SiO$_2$ layers, a
separately prepared second Si substrate having a 500-nm
thick SiO$_2$ layer was superposed with the SiO$_2$ layer
inside, and the superposed matter was heat-treated at

600°C for 2 hours to bond the substrates tightly.

The porous layers were bared at the edge face of the wafer, and a liquid such as water was allowed to penetrate into the porous Si. The entire bonded wafer

5   was heated or cooled, whereby the porous Si layers were broken owing to expansion or other causes to allow the wafer to separate into three sheets with the porous Si layers exposed.

The porous Si layers were etched selectively

10   with a mixture of 49% hydrofluoric acid with 30% hydrogen peroxide (1:5) with agitation. Thereby the porous Si was etched selectively and removed completely with the monocrystalline Si remaining unetched as an etch-stopping material. The etching rate of the

15   nonporous monocrystalline Si was extremely low, the selection ratio of the etching rate of the porous Si being 10^5 or higher. Therefore, thickness decrease of the nonporous layer by etching was practicably negligible (several ten Å).

20   Consequently, a monocrystalline Si layer was formed in a thickness of 1 μm respectively on the two Si oxide films simultaneously. The monocrystalline Si layers did not change at all by the selective etching of the porous Si layer.

25   The cross-section of the Si layer was observed by transmission electron microscopy, and it was confirmed that no additional crystal defect was formed

in the Si layer and the excellent crystallinity was retained.

The same results were obtained without formation of the oxide film on the surface of the epitaxial Si layer.

The first monocrystalline Si substrate was used repeatedly for the same use after removal of the remaining porous Si and flattening of the surface by hydrogen treatment.

Example 11

A first monocrystalline (100) Si substrate of P-type or N-type having a diameter of 5 inches, a thickness of 625 μm, and a specific resistance of 0.01 Ω•cm was anodized in an HF solution under the anodization conditions below:

Current density: 7 mA•cm^{-2}

Anodization solution: $HF:H_2O:C_2H_5OH = 1:1:1$

Time: 4 minutes

Thickness of porous Si: 3 μm

Porosity: 15 %

The anodization was conducted further under the conditions below:

Current density: 30 mA•cm^{-2}

Anodization solution: $HF:H_2O:C_2H_5OH = 1:3:2$

Time: 3 minutes

Thickness of porous Si: 10 μm

Porosity: 45 %

This substrate was oxidized at 400°C in an oxygen atmosphere for one hour. Thereby the inner wall of the pores of the porous Si was covered with a thermal oxidation film. On the porous Si formed on the substrate, monocrystalline Si was allowed to grow epitaxially in a thickness of 0.3 µm by CVD under the conditions below:

Source gas: SiH_4

Carrier gas: H_2

Temperature: 850°C

Pressure: 1×10^{-2} Torr

Growth rate: 3.3 nm/sec

The surface of the formed epitaxial Si layer was thermally oxidized to form SiO_2 layer in a thickness of 100 nm.

On the face of the SiO_2 layer, a separately prepared second Si substrate having a 500-nm thick SiO_2 layer was superposed with the SiO_2 layer inside, and the superposed matter was heat-treated at 700°C for 2 hours to bond the substrates tightly.

A force was applied to the first (or second) substrate in a direction parallel to the second (or first) substrate, whereby the porous Si layer was broken by the shear stress to allow the wafer to separate into two sheets with the porous Si layers exposed.

The porous Si layer was etched selectively with

an HF/HNO$_3$/CH$_3$COOH type etching solution. Thereby the porous Si was etched selectively and removed completely. The etching rate of the nonporous monocrystalline Si was extremely low, so that the

5   thickness decrease of the nonporous layer by etching was practicably negligible.

Consequently, a monocrystalline Si layer was formed in a thickness of 1 μm on the Si oxide layer. The monocrystalline Si layer did not change at all by

10   the selective etching of the porous Si layer.

The cross-section of the Si layer was observed by transmission electron microscopy, and it was confirmed that no additional crystal defect was formed in the Si layer and the excellent crystallinity was

15   retained.

The same results were obtained without forming the oxide film on the surface of the epitaxial Si layer surface.

The first monocrystalline Si substrate was used

20   repeatedly for the same use after removal of the remaining porous Si.

Example 12

A first monocrystalline (100) Si substrate of P-type or N-type having a diameter of 5 inches, a

25   thickness of 625 μm, and a specific resistance of 0.01 Ω•cm was anodized in an HF solution under the anodization conditions below:

Current density: 7 mA·cm^{-2}

Anodization solution: HF:H$_2$O:C$_2$H$_5$OH = 1:1:1

Time: 4 minutes

Thickness of porous Si: 3 μm

5   Porosity: 15 %

The anodization was conducted further under the

conditions below:

Current density: 30 mA·cm^{-2}

Anodization solution: HF:H$_2$O:C$_2$H$_5$OH = 1:3:2

10   Time: 3 minutes

Thickness of porous Si: 10 μm

Porosity: 45 %

This substrate was oxidized at 400°C in an

oxygen atmosphere for one hour. Thereby the inner wall

15   of the pores of the porous Si was covered with a

thermal oxidation film. On the porous Si formed on the

substrate, monocrystalline Si was allowed to grow

epitaxially in a thickness of 0.3 μm by CVD under the

conditions below:

20   Source gas: SiH$_4$

Carrier gas: H$_2$

Temperature: 850°C

Pressure: 1 × 10^{-2} Torr

Growth rate: 3.3 nm/sec

25   The surface of the formed epitaxial Si layer

was thermally oxidized to form SiO$_2$ layer in a thickness

of 100 nm.

On the face of the SiO_2 layer, a separately
prepared second Si substrate having a 500-nm thick SiO_2
layer was superposed with the SiO_2 layer inside, and the
superposed matter was heat-treated at 700°C for 2 hours
5   to bond the substrates tightly.

The porous layers were bared at the edge face
of the wafer, and the porous Si was etched from the
edge face with a selective etching solution, whereby
the wafer came to be separated into two sheets.

10   Further, the porous Si layer on the second Si
substrate was etched selectively with an $HF/HNO_3/CH_3COOH$
type etching solution. Thereby the porous Si was
etched selectively and removed completely. The etching
rate of the nonporous monocrystalline Si was extremely
15   low, so that the thickness decrease of the nonporous
layer by etching was practicably negligible.

Consequently, a monocrystalline Si layer was
formed in a thickness of 1 μm on the Si oxide film.
The monocrystalline Si layers did not change at all by
20   the selective etching of the porous Si layer.

The cross-section of the Si layer was observed
by transmission electron microscopy, and it was
confirmed that no additional crystal defect was formed
in the Si layer and the excellent crystallinity was
25   retained.

The same results were obtained without forming
the oxide film on the surface of the epitaxial Si layer

surface.

The first monocrystalline Si substrate was used repeatedly for the same use after removal of the remaining porous Si.

WHAT IS CLAIMED IS:

1.   A process for producing a semiconductor substrate comprising steps of:

forming a nonporous monocrystalline semiconductor layer

5   on a porous layer of a first substrate having the

porous layer;

bonding the nonporous monocrystalline layer onto a

second substrate;

separating the bonded substrates at the porous layer;

10   removing the porous layer on the second substrate; and

removing the porous layer constituting the first

substrate.

2.   A process for producing a semiconductor

15   substrate, comprising steps of:

forming a nonporous monocrystalline semiconductor layer

on a porous layer of a first substrate having the

porous layer;

bonding the nonporous monocrystalline layer onto a

20   second substrate with interposition of an insulative

layer;

separating the bonded substrates at the porous layer;

removing the porous layer on the second substrate; and

removing the porous layer constituting the first

25   substrate.

3.   The process according to claim 1 or 2,

wherein the porous layer is formed from silicon.

4.   The process according to claim 1 or 2,
wherein on the separated first substrate, after removal
of the porous layer therefrom, a new porous layer is
formed, and is employed repeatedly as the first
substrate in the forming step of the nonporous
monocrystalline semiconductor layer and subsequent
steps.

5.   The process according to claim 1 or 2,
wherein the nonporous crystalline semiconductor layer
is an Si layer.

6.   The process according to claim 1 or 2,
wherein the nonporous crystalline semiconductor layer
is a compound semiconductor layer.

7.   The process according to claim 1 or 2,
wherein the first substrate is constituted from Si.

8.   The process according to claim 1 or 2,
wherein the second substrate is light-transmissive.

9.   The process according to claim 1 or 2,
wherein the step of removing the porous layer is
conducted by etching.

10.   The process according to claim 1 or 2, wherein the step of removing the porous layer is conducted by selective grinding of the porous layer by employing the nonporous monocrystalline semiconductor layer as a stopper.

11.   The process according to claim 1 or 2, wherein the step of separating the bonded substrates at the porous layer is conducted by at least one of methods of application of a compression force to the substrate in a direction perpendicular to the bonding face of the substrate, application of a pulling force to the substrate in a direction perpendicular to the bonding face of the substrate, and application of a shear stress to the bonding face.

12.   The process according to claim 2, wherein the insulative layer is formed on at least one of the nonporous monocrystalline layer and the surface of the second substrate.

13.   The process according to claim 12, wherein the insulative layer is selected from thermal oxidation films, deposited SiO_2 films, and deposited Si_3N_4 films.

14.   The process according to claim 1 or 2,

wherein the step of bonding is conducted one or combination of anode coupling, compression, and heat treatment.

5   15.   The process according to claim 1 or 2, wherein the porous layer is formed by anodization.

16.   The process according to claim 1 or 2, wherein the anodization is conducted in an HF solution.

10   17.   The process according to claim 1 or 2, wherein the step of separating the substrates at the porous layer is conducted by application of a wave energy.

15   18.   The process according to claim 1 or 2, wherein the step of separating the substrates at the porous layer is conducted by inserting a separation member from an edge face of the porous layer thereinto.

20   19   The process according to claim 1 or 2, wherein the step of separating the substrates at the porous layer is conducted by expansion energy of a material impregnated into the porous layer.

25   20.   The process according to claim 1 or 2, wherein the step of separating the substrates at the

porous layer is conducted by selective etching at the
edge face of the wafer.

21.   The process according to claim 1 or 2,
5   wherein the porosity of the porous layer ranges from 10
to 80 %.

ABSTRACT OF THE DISCLOSURE

A process for producing a semiconductor substrate is provided which comprises steps of forming a porous layer on a first substrate, forming a nonporous monocrystalline semiconductor layer on the porous layer of the first substrate, bonding the nonporous monocrystalline layer onto a second substrate, separating the bonded substrates at the porous layer, removing the porous layer on the second substrate, and removing the porous layer constituting the first substrate.

COMBINED DECLARATION AND POWER OF ATTORNEY
FOR PATENT APPLICATION

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name;

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled ___PROCESS FOR PRODUCTION OF___ SEMICONDUCTOR SUBSTRATE _____, the specification of which

☐ is attached hereto.   ☒ was filed on __March 9, 1995_____ as Application Serial No. 08/401,237_____

and was amended _____ (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, §1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

Country	Application No.	Filed (Day/Mo./Yr.)	Priority Claimed (Yes/No)
JAPAN	6-39389	10 March 1994	YES
JAPAN	7-45441	6 March 1995	YES

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FOR PATENT APPLICATION

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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FIG. 1A
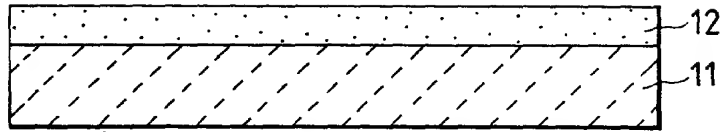
FIG. 1B
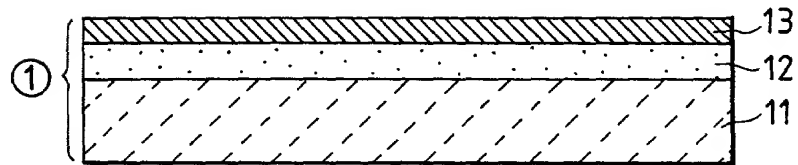
FIG. 1C
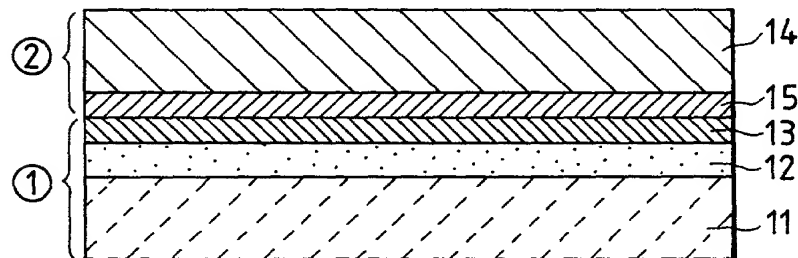
FIG. 1D
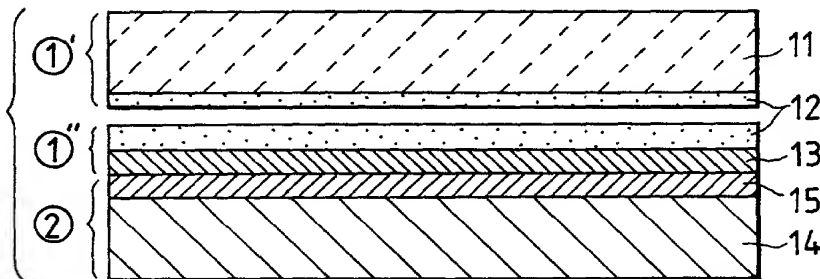
FIG. 1E
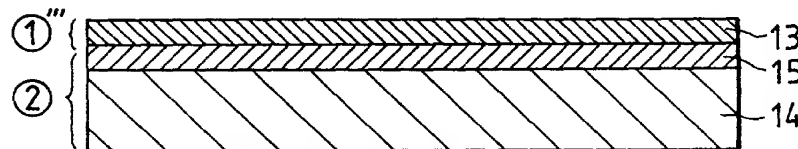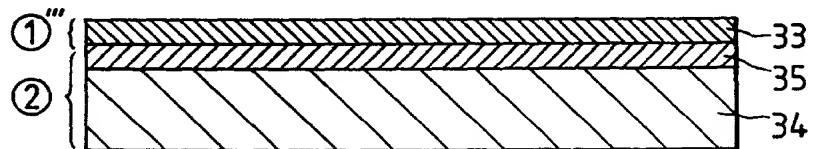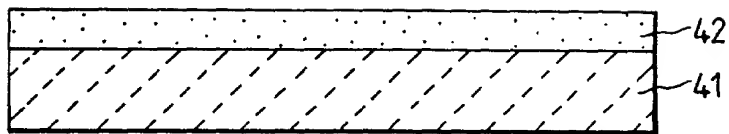
FIG. 2A
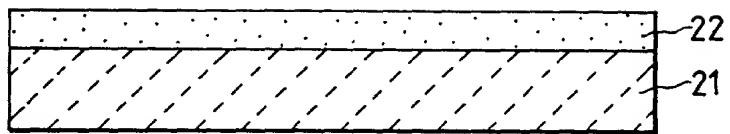
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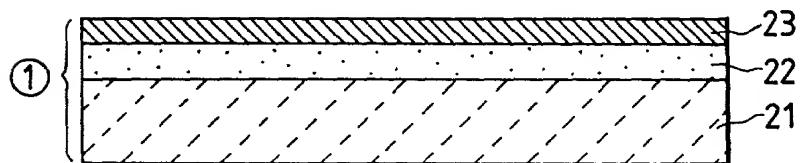
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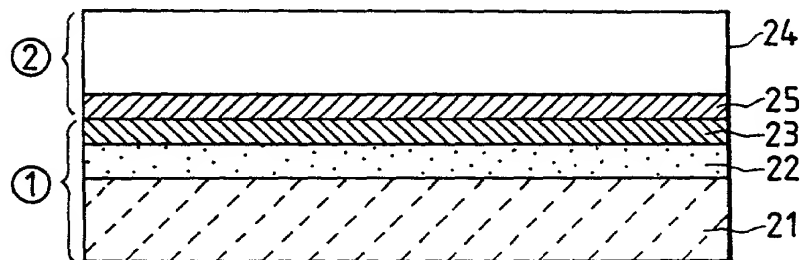
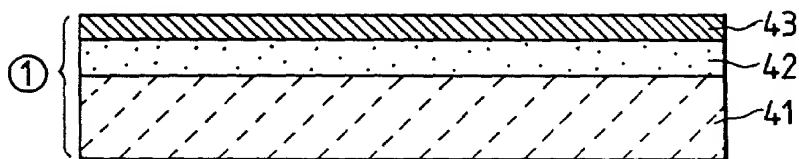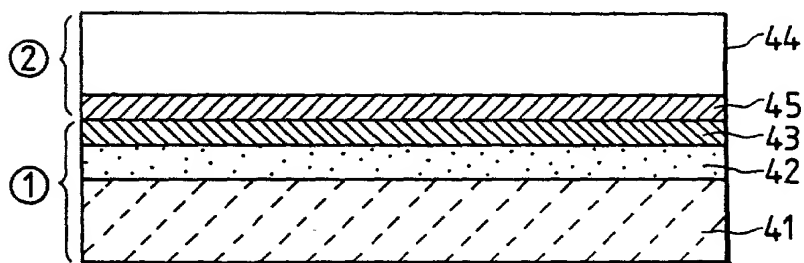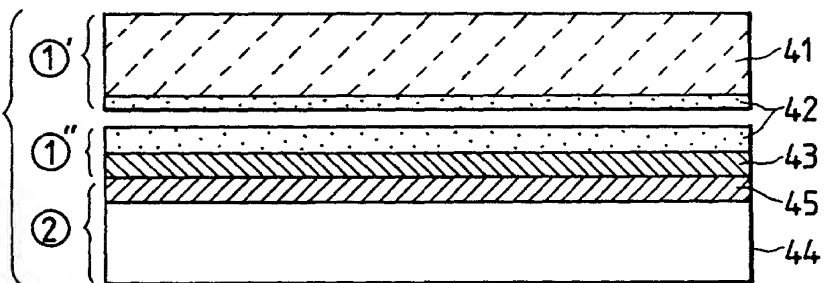FIG. 2D
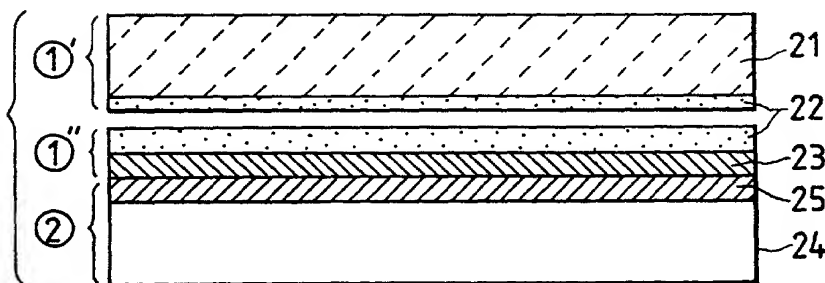
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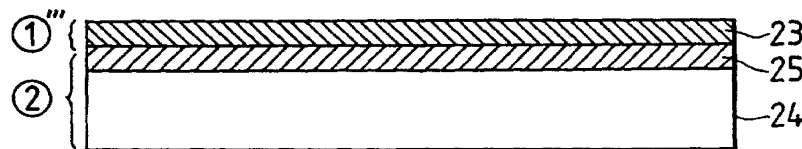
FIG. 3A
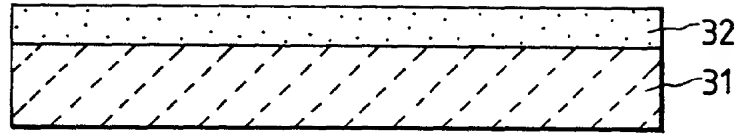
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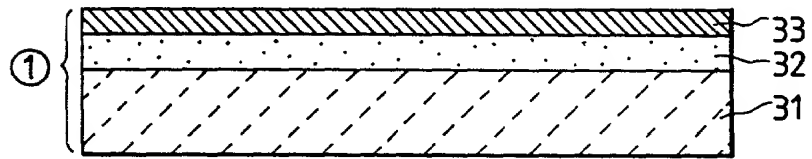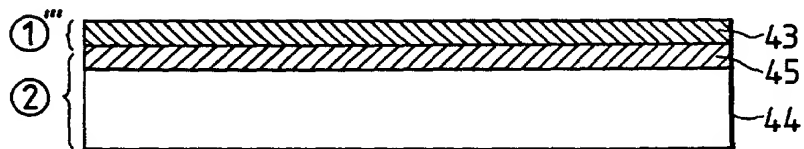
FIG. 3C
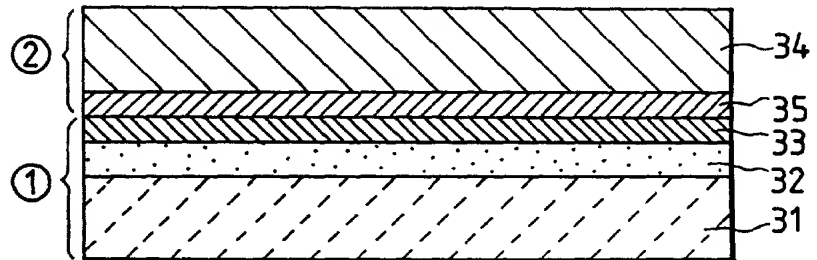
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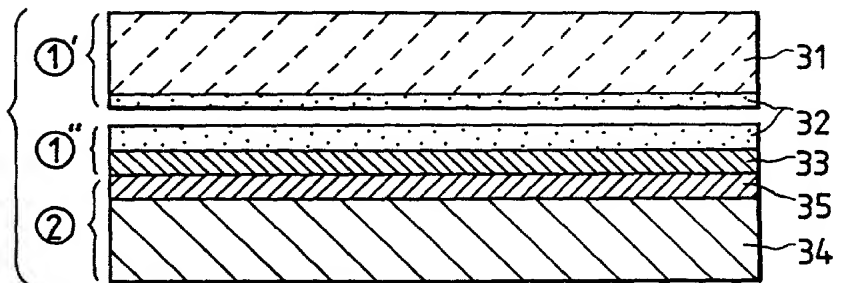
FIG. 3E

FIG. 4A

FIG. 4B

FIG. 4C

FIG. 4D

FIG. 4E

FIG. 5A

FIG. 5B

FIG. 5C

FIG. 5D

FIG. 5E